FOOD TECHNOLOGY

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January, 1953 Pages 1-48 Volume 7, Number 1

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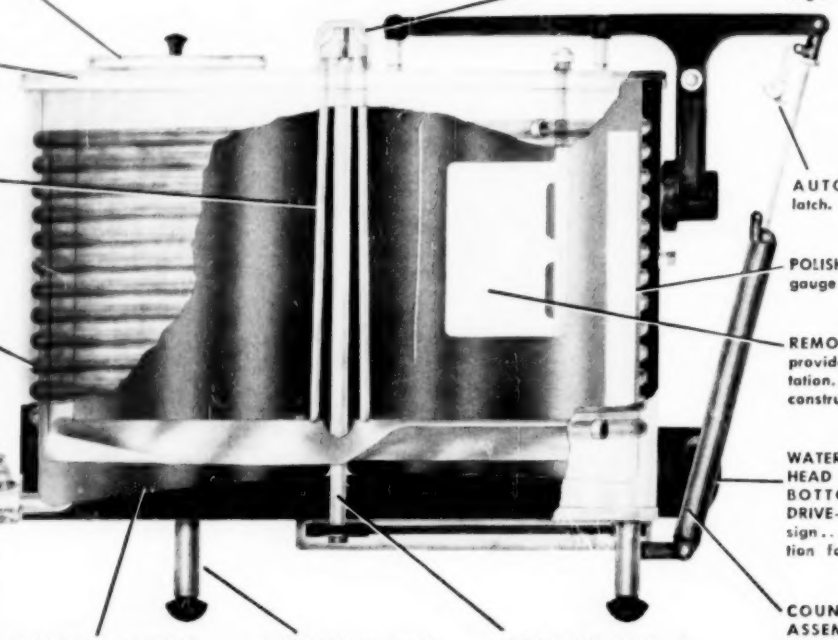

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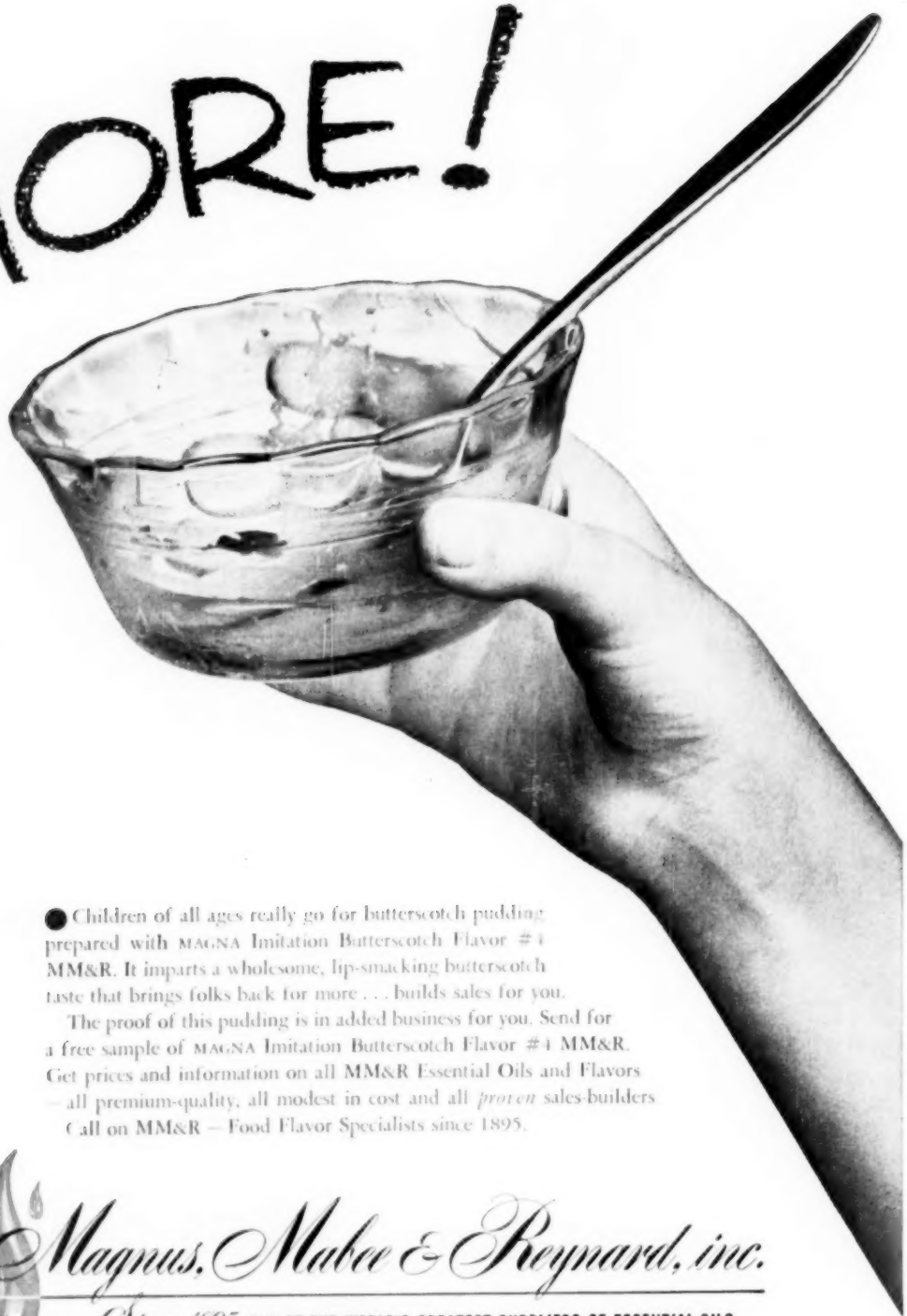

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The Institute of Food Technologists assumes no responsibility for statements in contributed articles appearing in FOOD TECHNOLOGY. Editorials present only opinions of the writers and do not necessarily reflect policies of the Institute of Food Technologists.

Affairs of the Institute

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News of the Regional Sections
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BOSTON MEETING TO BE OUTSTANDING

If planning and foresight are any criteria, the 1953 IFT Convention promises to be one of the outstanding events in many years. Even before the last convention, General Chairman Loren B. Sjöström had started his preparation. Starting with Honorary Chairman Professor Bernard E. Proctor, all the necessary committees have been arranged and have been functioning. According to Charles H. Shackelford of the Great Atlantic & Pacific Tea Company's National Fish Department, Chairman of the Exhibit Committee, forty exhibits have already been sold, and if your company wants to be present at this occasion, it has been requested that you ask that they get in an application promptly because the space is definitely limited.

The Program Committee, under Chairman J. Avery Dunn of the Atlantic Gelatin Division of General Foods Corporation, has been hard at work and the over-all program is shaping up rapidly. The Executive Council will meet on Saturday, June 20, with the Editorial Board Meeting Saturday evening. On Sunday, the National Council meets, with a mixer to be held Sunday evening. This will be complimentary and ladies are invited to attend.

Program Notes

Monday morning starts the General Session, and in addition to the annual President's address there will be featured speakers including Dr. Karl Compton. Other speakers have not been confirmed so far. On Monday noon there will be a luncheon at which the Babcock and the Food Engineering awards will be presented.

On Monday afternoon there will be a very interesting session on the general subject of geriatric foods. This is of particular importance because the proportion of our population over the age of 60 is already large and is increasing at a very rapid rate. The wide-awake food processor will do well to give consideration to the problem of supplying the right type of food to this group which, incidentally, is relatively high in income. This program, being developed by Dr. Charles Glenn King and Dr. R. C. Sherwood, should be one of the highlights of the entire meeting.

On Tuesday, both morning and afternoon sessions, there will be a fish symposium under the leadership of Dr. W. F. Hampton and Dr. Hugh L. A. Tarr. Practical and technical subjects will be discussed. When the IFT met in Boston previously, the fish session was very well attended and evoked a great deal of interest.

Also on Tuesday, in a morning session, there will be a program on fermented products, under the direction of Dr. Fred W. Fabian. A session on chemicals in food under the direction of Dr. Charles N. Frey is also scheduled for Tuesday morning. This continues to be a subject of the greatest interest to food processors.

On Wednesday morning there will be a packaging symposium which is being developed by Dr. L. W. Elder who has spent a good many years developing and improving packages for foods. Another Wednesday event—with both a morning and afternoon session—is the program on canning and processing technology which is being developed by Dr. E. J. Cameron and Dr. C. Olin Ball. During this session the Appert Medal Address will be presented. On Wednesday, also—at the morning and afernoon sessions—there will be a program on research in the field of fruit and vegetable technology under the direction of Dr. Emil M. Mrak.

Banquets, Luncheons, and Group Meetings

The Banquet and Luncheon Committee, under the chairmanship of John H. Moriarty of Arthur D. Little Company, has scheduled a complimentary luncheon on Sunday, June 21, for the Council. On Monday noon the Babcock and the Food Engineering Awards will be presented. Tuesday evening, June 23, the Appert Medal will be presented at a dinner to which ladies are invited.

As usual there will be breakfast and luncheon meetings for the alumni of various colleges and universities. There will be a sectional officers breakfast on Monday morning, June 22. The University of Massachusetts breakfast comes on Monday morning, the M.I.T. breakfast on Tuesday morning, and the Canadian breakfast on Wednesday morning.

Dr. Dunn has suggested the desirability of holding special group meetings on Saturday and Sunday preceding the general meetings. The holding of these special committee meetings during the pre-convention days has always resulted in considerable conflict and has prevented members from attending the sessions because they felt obligated to attend the special meetings. Plans have been made whereby small meeting rooms for these special groups can be made available on both Saturday and Sunday. If any groups planning such meetings, would like to take advantage of this opportunity, they are urged to get in touch with Dr. Dunn immediately.

To the Ladies

On the distaff side, Chairman Ruth M. Powers of the Arthur D. Little Company has developed a round of entertainment for the ladies which should make the visit of wives of members something to be remembered. Nothing is being left undone to make this a memorable occasion. Members are urged to call this section of this report to the attention of their wives. Starting Monday, June 22, there is to be a sightseeing tour of the North Shore. This is an all-day event and will include lunch at some traditional place at Rockport. This is entirely complimentary, including the lunch. On Tuesday afternoon there will be another tour which includes a concert and tea at the Isabella Stewart Gardner Museum. This, too, is complimentary to all ladies attending. Wednesday will be devoted to shopping tours. This is another all-day session and includes the famous Chestnut Hill Shopping Center and the Shoppers' World. For this occasion free transportation will be provided.

Miss Powers points out that, through the courtesy of Mary Cremmen of the Boston *Globe*, a complete list of

(Continued on page 6)

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Boston Meeting to be Outstanding

(Continued from page 4)

what to wear for this time of the year has been prepared. Copies are available by writing Miss Powers. It gives all the clothing needed, as picked out by outstanding fashion editors.

There is available an interesting 50-page booklet entitled, "Fascinating Trips to Historic Spots In and About Boston." These booklets may be had by writing to Miss Mary Harrigan of the Publicity Committee. She can be addressed in care of the U. S. Food and Drug Administration, Department of Agriculture, Federal Security Agency, 408 Atlantic Ave., Boston.

Since Boston and environs boast of 200 different universities, colleges and schools, many members attending may wish to use their occasion to visit them.

Mindful of the fact that "all work and no play makes John a dull boy," the committee has again arranged to take over the Boston Symphony Hall at a Pops Concert on Monday evening, June 22. Included will be a pitcher of the famous Pops Punch. This is an occasion that few will want to miss, and ladies as usual are invited.

Clambake!

The main relaxation comes on Wednesday, June 24, at the close of the afternoon session. This includes a clambake which will be held in the Rockwell Cage at M.I.T. Catered as only the famous Seiler's can do it, this clambake will be followed by a barn dance in the evening. Everyone is warned to bring dungarees or other suitable informal clothes to relax in a good old-fashioned New England way. This time there are no boats or stormy oceans to upset the evening's entertainment.

Thursday is to be devoted to plant trips. This is under the chairmanship of Ernest C. Peakes of the New England Confectionery Company and will cover a wide range of industrial plants. If you have any special interest, it is suggested that you write Mr. Peakes your wishes.

Members of the Institute of Food Technology can be a real help to the Committee by getting their reservations in early. Everything has been arranged for your convenience and edification, and a real New England welcome awaits you. The place to be in '53 is at the Boston Convention.

Regional Section News

AMES SECTION

On October 24, 1952, the first meeting in the Pine Room, Memorial Union, Iowa State College, was devoted to talks on recent developments in the canning industry. Dates and topics of other meetings to be held at the Memorial Union, Iowa State College, are:

Friday, January 30, 1953—Packaging of Foods.

Friday, April 24, 1953—Drying and Condensing of Foods.

On Friday, October 24, 1952, the following talks were heard:

"Recent Advances in Canning Equipment and Processing." John J. Toney, Chemist, Food Machinery and Chemical Corporation, Hoopeston, Illinois.

"New Applications of Dairy Processing Equipment to Other Branches of the Food Industry." Eugene O'Neil, Cherry-Burrell Co., Cedar Rapids, Iowa.

Mr. M. L. Schumacher, Morrell Packing Co., Ottumwa, Iowa, gave a most interesting report about his recent trip to Sweden.

Mr. C. W. Swanson, Sales Engineer, Minneapolis-Honeywell Regulator Co., Brown Instruments Division, was the dinner speaker. He gave an illustrated talk on "Instrumentation, the Plus Factor in Modern Processing."

NORTHERN CALIFORNIA SECTION

Robert Berry, American Home Foods, Inc., has been elected 1953 chairman of the Northern California Section, Institute of Food Technologists. The election of Berry—who succeeds Richard D. McKirahan of American Can Co.—was announced at the annual meeting of the section, held in Albany and Oakland on December 4.

Harold Olcott, of USDA's Western Regional Research Laboratory, is the new 1953 vice-chairman. Elise Hoffman, Golden State Co., has been named secretary, and Charles P. Collier, National Canners Assn., treasurer. Elected to serve on the executive committee are Gordon MacKinney and John Kilbuck, both of University of California. Hugh T. Griswold, Owens-Illinois Glass Co., was re-elected to serve as national councillor.

The daytime program for the annual meeting, sessions of which were held at USDA's Western Regional Research Laboratory in Albany, consisted of contributed papers followed by discussions. The subject of each paper, the author, and the discussion leader, were as follows:

"Recent Developments in the Manufacture of 36% Tomato Paste," by Geo. D. Armerding, Mojonnier Bros. Co. Edwin L. Mitchell, Richmond-Chase Co., discussion leader.

"Organoleptic Investigations of Pesticide-treated Fruit and Vegetable Crops," by Elly Hinreiner, and Marion Simone, University of California. Frank Lamb, National Canners Assn., discussion leader.

"A Preliminary Theory of Batch Drying in the Low-moisture Range," by W. B. Van Arsdel, U. S. Dept. of Agriculture. William V. Cruess, University of California, discussion leader.

"Tenderometer Measurements in Cooked Product for Estimating Maturity of Frozen Peas," by Rachel Makower, U. S. Dept. of Agriculture. Shirley Volmer, Western Beet Sugar Producers, discussion leader.

"Temperature of Fluids in Transit through Tubular Heaters and Coolers," by Russell L. Perry, University of California. William McK. Martin, Dole Engineering Corp., discussion leader.

"Chlorination as Applied to Sanitation in a Fruit Cannery," by Bruno A. Filice, Filice & Perrelli Can-

(Continued on page 8)

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Regional Section News

(Continued from page 6)

ning Co. Robert Quirk, Barron-Gray division, Hawaiian Pineapple Co., discussion leader.

"Distribution of Amino Acids in Citrus Fruit Tissues," by Phillip M. Townsley and Maynard A. Joslyn, University of California. Gordon MacKinney, University of California, discussion leader.

"Effect of Syrup Volume and Density on Browning of Frozen Peaches," by Dante G. Guadagni, U. S. Dept. of Agriculture. Harry Pancoast, American Maize-Products Co., discussion leader.

"Neglected Chemicals in Foods," by Floyd de Eds, U. S. Dept. of Agriculture. Louis G. Petree, Continental Can Co., discussion leader.

"Development of Oxidative Rancidity of Meat Fats during Freezing Storage," by Aloys L. Tappel, University of California. Harold S. Olcott, U. S. Dept. of Agriculture, discussion leader.

"Effect of Cooking on the Thiamine Content of Brown Rice," by Barbara M. Kennedy, University of California. Helen Hanson, U. S. Dept. of Agriculture, discussion leader.

"Alcohol Accumulation in Underscalded Frozen Broccoli," by Paul A. Buck and Maynard A. Joslyn, University of California. Calvin Bright, Stokely Foods, Inc., discussion leader.

Maynard Joslyn served as chairman of the morning session, and Harold Olcott in the afternoon.

In the evening the section met for dinner in Oakland, after which door prizes were distributed and an interesting discussion of the work of U. S. Food and Drug Administration was presented by McKay McKinnon, chief of the San Francisco district operations of the federal agency.

CHICAGO SECTION

The first Fall Meeting of the Chicago Section was held in the Palmer House on Monday November 10, 1952. Speaker of the evening was Dr. Randall Royce, Assistant Manager of the Central Division Laboratory, American Can Company. Dr. Royce has been associated with this company since receiving his Ph.D. degree in 1934 from the University of Kansas. He specializes in the improvement of canning, freezing and other methods of preserving foods as well as in the development of food containers and methods of handling.

Dr. Royce pointed out that the present tin can is a far cry from the original tin can developed by Durand in 1810 and used by Appert in his historic work on the preservation of foods by canning. But even today the can companies are carrying on extensive research programs on the development of more satisfactory containers with particular emphasis on the reduction of critical raw materials such as tin.

Various technical terms used in tin can manufacture were explained. Substitute metal such as aluminium for bodies, and various types of solders, enamels and sealing compounds were described.

An event of the evening was the formal presentation of the First Annual Monsanto IFT Award to Dr. Richard Forsyth for delivery of the best technical paper given at the Annual National Meeting of the IFT in Grand Rapids last June. The award was presented by Charles S. Lawrence, Executive Secretary of IFT who explained the extreme care used in selection of the awardee.

MARYLAND SECTION

The November meeting of the Maryland Section was held at the Hotel Stafford, Baltimore, Maryland, on November 14, 1952, and took the form of a symposium devoted to the discussion of objective methods of measuring the quality of foods. The symposium started at 1:00 p.m. and was divided into two sessions. Subjects discussed and speakers taking part in the symposium were as follows:

"Filth as a Measure of Quality in Food Products." Edward M. Hoshall, Chief Chemist, Food and Drug Administration, Baltimore.

"Objective Methods of Measuring Quality in Poultry Products." A. W. Brant, Food Technologist, Bureau of Animal Industry, U. S. Department of Agriculture, Beltsville, Md.

"Objective Methods for Determining the Quality of Fishery Products." H. W. Nilson, Pharmacologist in Charge, U. S. Fish and Wildlife Service, College Park, Md.

"Judging the Quality of Dairy Products." W. S. Arbuckle, Professor of Dairy Manufacturing, University of Maryland, College Park, Md.

"The Peroxidase Test as a Measure of Fresh Pack Pickle Quality." Arthur M. Kaplan, Food Bacteriologist, Bureau of Human Nutrition and Home Economics, U. S. Department of Agriculture, Beltsville, Md.

"Objective Methods for Flavor Evaluation." John Fromm, Quality Supervisor, The Calvert Distilling Co., Relay, Md.

"The Dulaney Approach to Quality Control." William J. Hart, Jr., Chief Technologist, John H. Dulany and Son, Inc., Fruitland, Md.

"Objective Methods Used in the Fruit Processing Industry." J. R. Cogley, Food Technologist, Knouse Foods Cooperative, Peach Glen, Pa.

"The Work of the IFT Committee on Objective Methods." B. W. Clarke, Chief Food Technologist, Crosse and Blackwell, Baltimore, Md.

"Objective Methods for Measuring Kinesthetic Factors of Quality in Foods." Amihud Kramer, Professor of Horticulture, University of Maryland, College Park, Md.

"Comparison of Objective Methods for the Evaluation of Baked Cereal Products and Their Ingredients, and Their Relation to Quality Considerations." S. W. Arenson, Director, Food Development Laboratories, Baltimore, Md.

(Continued on page 10)

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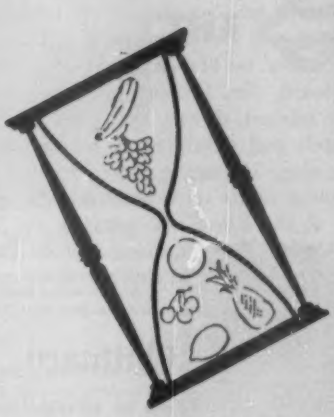

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Regional Section News

(Continued from page 8)

Dessert for the dinner consisted of pumpkin pies made from canned pumpkin provided by W. L. Wheatley Inc., Clayton, Delaware. Members and guests served as a judging panel for the pies.

WESTERN NEW YORK SECTION

The Buffalo Area Group of the WNY Section heard Mr. Clarence Sorg, Jr., of the International Salt Co. address its December 3rd meeting in the Kathryn Lawrence Dining Room, 641 Delaware Ave., Buffalo, N. Y. Mr. Sorg spoke on the mining and refining of salt and its uses in the food industries.

NORTHEAST SECTION

On December 4th the Section met in the Hotel Brunswick, Boston, Mass., for dinner and to hear Dr. Bill Hampton, Director of the Fisheries Research Laboratory, Birdseye Division, General Foods Corp., Boston, discuss "The Production of Frozen Fish Fillets." He illustrated his talk. In addition, two films in colour obtained through the courtesy of the Esso Standard Oil Company were shown. One, "Rendezvous in the Reef," was all about fish; the other, "Island Oddities," portrayed sea-bird life.

OREGON SECTION

The Section held its December meeting on the 4th at the National Biscuit Company, 100 N. E. Columbia Blvd., Portland, Ore. The meeting took the form of an afternoon plant tour. The Nabisco plant is one of the largest, most modern bakeries in this area, and represented a branch of the food industry which the Oregon Section, as a group, had not had occasion to visit in the past.

The Ernest H. Wiegand Student Chapter has held recent meetings on October 7 and November 6. Mr. J. Roger Deas, Manager of the Public Information Service of the American Can Company, addressed the former meeting on the progress of can-making technology. The meeting was also attended by the Chapter's Dairy Club and was held in Withycombe Hall, the new dairy building. The November 6 meeting was put on by the Food Technology staff, purpose of which was to familiarize the students with the research now in progress in the Food Technology Department. Next meeting is slated for January.

PERSONNEL

The University of Buffalo has introduced a new course, Chemical Marketing, being offered for graduate credit in the evening session during the 1952-1953 sessions. The course, jointly sponsored by the Departments of Chemistry and Marketing, is being given by Dr. Frank P. Greenspan of the Research and Development Department of the Buffalo Electro-Chemical Company, Inc.

Curtis E. Maier, general manager of research for the Continental Can Co., Chicago, was named chairman of Illinois Institute of Technology's Food Engineering Council at the recent biennial meeting of that group. Mr. Maier, who has been with Continental Can since 1937, succeeds Dr. Paul V. Manning, vice president in charge of research for International Minerals and Chemicals Corporation, Chicago. He will be the third chairman of the group.

At the suggestion of Professor Milton E. Parker, director of I. I. T. food engineering, the new chairman is to appoint four committees to: (1) Evaluate methods and equipment used in food preparation; (2) Study the professional status of food engineering; (3) Advise on methods to encourage industrial research in food engineering; (4) Advise on methods of encouraging young men to seek careers in food engineering.

John T. Knowles, vice president of Libby, McNeill & Libby, Chicago, said at the meeting that increased use of mass production methods in the food processing industry has pointed up the great demand for food engineers. It was reported that the average 1949 Illinois Tech food engineering graduate is now earning $5,500 per year. Dr. Berton S. Clark, scientific director for the American Can Company, Chicago, recommended that special efforts be initiated to advise executives of the potential pool of food engineers being provided by I. I. T. Mr. F. W. Specht, the president and chairman of the board for Armour and Company, and a guest at the meeting, said that business leaders are becoming more appreciative of the need for funds for training and research because of daily advances in this field.

Dr. L. E. Clifcorn, director of Continental Can Company's fundamental research, has been appointed chairman of the Panel on Food of the Committee on Equipment and Supplies of the Department of Defense Research and Development Board, according to an announcement by Mr. Walter G. Whitman, chairman of the Board. Dr. Clifcorn will head the five-man panel which advises, directs, and gives technical guidance on research and development related to the production, processing, packaging, and service of food, with the exception of its nutritional aspects, which is the concern of the Board's Committee on Medical Sciences. Mr. Paul S. Forsyth, director of the Panel on Food, will assist Dr. Clifcorn in this part-time assignment.

Obituary

It is with sorrow that we record the death on November 23, 1952, of Joseph Francis Rudolph, President of Dodge and Olcott, Inc. He was a loyal friend and supporter of the Institute and of this journal and was widely known and respected among the membership.

This, we believe...

A *good* name is the greatest reward any man or company can attain . . .

A *good* name is honor, character, integrity and pride—in every product bearing that name, in all dealings under that name . . .

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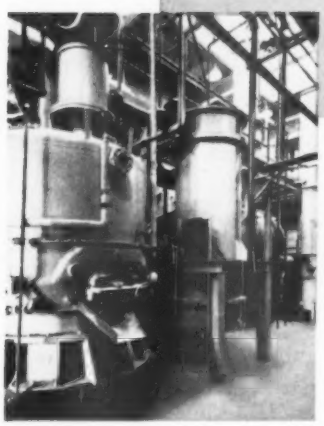

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Books

SYMPOSIUM ON RADIOBIOLOGY. Edited by James J. Nickson, M.D. 465 pages, John Wiley and Sons, Inc., New York. Price $7.50.

This book, composed of twenty-three papers delivered before the Oberlin Symposium on Radiobiology in June of 1950, is devoted to studies on the fundamental effects of radiations on matter considered from a biological viewpoint—as the title suggests. The symposium was sponsored by the Subcommittee on Radiobiology of the Committee on Nuclear Science of the National Research Council, assisted by the Atomic Energy Commission and the Office of Naval Research. The objective of the symposium was a "thorough examination of the fundamental concepts that exist in radiobiology."

The editorial committee responsible for putting the manuscripts in shape for book publication represent the leaders in the field of radiobiology and associated subjects as do the authors of the various papers.

This book is to be strongly recommended as background reading for persons interested in the potential application of ionizing radiations to the sterilization of foods and heat-sensitive materials and is recommended as well to those who are directly interested in radiobiology.

The chapters by Platzman and Burton present the theoretical and basic physical-chemical effect of radiations; those by Dale and Guzman-Barron treat the chemical effects of biological materials.

Physical processes are thoroughly covered in chapters by Morrison, Fano, Wilson, Solomon and others. Biological effects are well described by Miller, Zirkle, Tobias, Evans, Jones, Russell, Brues, and Sacher. The chapters by Hollaender and Laterjet on the radiosensitivity of cells to ionizing radiations while very basic, have tremendous practical applications.

The text of the book is not free from some repetition, but this is difficult to avoid, of course, in a symposium of this sort. It is regrettable, too, that it took so long after the symposium to get the material published, but the path to the printer in publication enterprises of this kind is complicated. As the prefatory note explains "the vagaries of authors, publication committees, and the international situation" contributed to the delay.

Well bound and well printed on good paper, the book and its format are a credit to the art of modern printing. Since the book succeeds in bringing together fundamental concepts and ideas, which was its objective, it is also a credit to all who contributed to its accomplishment.

S. A. GOLDBLITH.

POLAROGRAPHY: Second completely revised and augmented edition in two volumes. I. M. Kolthoff and James J. Lingave. Interscience Publishers, New York, 1952. Volume II.

This volume is divided into four parts, as follows: part III, *ca* 200 pages, inorganic polarography; part IV, *ca* 200 pages, organic polarography; part V, 34 pages, biological applications; part VI, 66 pages, amperometric titrations. Much of part IV is of biological interest, so that the number of pages allotted to part V might give an erroneous impression.

The work of the authors in the field of polarography and excellence of the first edition preclude the necessity for a formal evaluation of the present volume.

The revised edition is authoritative and thorough. The rarity of an occasional trivial typographical error testifies to the care with which the book has been written. A minor criticism is that part V gave this reviewer a slight let-down. The title of chapter 46 raised some expectation concerning serological cancer detection, while the text, page 874, states rather offhandedly: "No doubt more systematic work is still necessary before the polarographic method can be used as a reliable test. . . ." Under a running title of "Catalytic waves of —SH containing compounds," this same chapter discusses waves for riboflavin, nicotinic acid and lipids, which is incongruous.

The criticism is essentially one of organization. It is admittedly a matter of opinion whether the *general* problem of catalytic waves, including the special case of cysteine, cystine, etc. might not logically have come after chapter 13, Volume I on catalyzed hydrogen waves. With equal logic, riboflavin and nicotinic acid might have appeared in chapter 45, heterocyclic nitrogen compounds. Chapter 46, then, appears somewhat disjointed and suffers by contrast with the excellence of the other chapters.

The purpose of this reviewer however is not to cavil at what is offered, but rather to point out that the food technologist cannot afford to ignore any important method that has applicability to foods. Applications are demonstrated in methods for Pb, Cu, Fe (including ferricyanide), SO_2, to mention but a few inorganic cases, as well as for a host of organic compounds. Nowhere else can one get a sounder appraisal of the potentialities of the method nor a more thorough survey of what has been accomplished than in these volumes by Kolthoff and Lingave.

GORDON MACKINNEY.

THE SCIENCE, RAW MATERIALS AND HYGIENE OF BAKING. By Alexander Urie and J. H. Hulse. 465 pages, MacDonald & Evans, Ltd., London, England 1952. Price (30s.)

The book is divided into five parts: Scientific Principles (114 pages), The Baking Processes (61 pages), Raw Materials: Their Origin, Nature and Composition (130 pages), Hygiene (85 pages), and Nutrition (41 pages). These five parts are subdivided into fifteen different sections with 23 plates, 50 figures, and 49 tables.

Although written by Englishmen who must work with ingredients, especially flour, somewhat different from ours, this book is sufficiently broad to be of value to American students of baking, baking technicians, food chemists, and production men of the baking industry. There are not too many good texts relating to the science of baking and this book is a worthwhile addition to the literature.

(Continued on page 14)

FELCAFE, FELTON'S NEW COFFEE IMITATION, ACCLAIMED COAST TO COAST!

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As a result of several years of research and development, Felton Chemical Company chemists have produced a new coffee flavor that has revolutionized the use of coffee in many products. Candy, ice cream and baked goods manufacturers have long been aware of the handicaps presented by the lack of stability of coffee flavor concentrates, and it was this problem which Felton tackled first. Literally hundreds of new chemicals were isolated in the research for proper ingredients, and at least fourteen new specific aromatics were developed.

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Felton's unusual flavor is non-volatile, and its stability eliminates any worry about cooking off or freezing out. Neither does it oxidize or become rancid.

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Felcafé retains its full-bodied flavor in Hard Candy, Toffee, Caramels, Fudge, Soft Centers, Creams and Fondants, Marshmallows, Gum, Starch Jellies, Chocolate.

Ideal for Foods and Desserts

Popular coffee flavor can now be successfully applied to Ice Cream, Ices and Sherbets, Packaged Ice Cream Mixes, Puddings, Syrups, Beverage Concentrates, Gelatin, Pie Fillings, Cake Fillings, Icings, Toppings, Sandwich Fillings, Carbonated Beverages, Extracts.

Versatility of New Flavor for Pharmaceuticals

Coffee as a flavor for pharmaceuticals can be a big factor in the success of all kinds of products such as Pharmaceuticals, Lozenges, Troches, Vitamin Tablets, Cough Syrup, Elixirs.

Write For Samples of This Amazing Coffee Flavor

Tell us about the products in which you would like to use Felcafé. We will be happy to send you directions and liberal samples for testing.

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Books

(Continued from page 12)

The book is without a bibliography, lack of which could be a handicap to the serious student of baking. The first part of the book—Scientific Principles, especially—could be difficult for the student and references to more complete texts would be helpful in gaining understanding. The promise of a broad study of the technology of baking, contained in the title, is completely fulfilled by this book. The typography is excellent and the binding is good.

E. C. EDELMANN.

Selected Abstracts*

ANALYTICAL METHODS

Paper chromatography of some starch conversion products.

BUCHAN, J. L., AND SAVAGE, R. I. *Analyst, 77,* 401-06 (1952).

Paper chromatography used to distinguish white malt syrup from liquid glucose. Propanol, ethyl acetate, water-6-1-3 ratio used as solvent. Spraying reagent 4% aniline in ethanol, 4% diphenylamine in ethanol-80% phosphoric acid, 5:5:1 ratio. Heating time 10 minutes at 80° C.

Determination of technical benzene hexachloride in peanuts and soils.

HORNSTEIN, I. *Anal. Chem., 24,* 1036 (1952).

A modification of earlier method to give more accurate values for peanuts.

Determination of water in alcohols.

WEST, P. W., SENISE, P., AND BURKHALTER, T. S. *Anal. Chem., 24,* 1250-2 (1952).

High-frequency oscillators for the direct measurement of the water content of monohydric and polyhydric ales.

Acid-base chemical indicators.

FISHER, H. A. *Mfg. Chemist, 23,* 319-22 (1952).

A review article on the types and range of acid-base chem. indicators including the mechanism of color change, routine analytic tests and applications of indicators to detergent testing, photography, medicine, soil testing.

Identification of organic compounds from infra-red spectra.

MARTIN, A. E. *Nature, 170,* 20-2 (1952).

The importance of extensive cataloging of the infra-red absorption spectra of org. compds. is emphasized. A standard exptl. procedure and a filing system is suggested.

BIOLOGICAL SCIENCES

BIOCHEMISTRY

Lipid metabolism in atherosclerosis.

ELLIS, F. W. *No. Carolina Med. J., 13,* 169, April 1952; *J. Am. Med. Assoc., 149,* 1602 (1952). Lipoprotein and lipids are transported in the serum as a finely dispersed stable "emulsion." Any disturbance in lipid metabolism (due to inadequate endocrine function) producing an aggregation of fat particles may lead to atherosclerosis. Certain atherosclerosis patients have benefited from a dietary restriction of fats.

Structure of proteins.

EDSALL, J. T. *Nature, 170,* 53-5 (1952).

Recent developments in the study of proteins and synthetic polypeptides are reviewed.

A comparison of dextran and oxypolygelatin in the treatment of hemorrhagic hypotension.

GROPPER, A. L. *Am. J. Physiol., 169,* 749-56 (1952).

Dextran and oxypolygelatin have been found to reduce mortality from 85% to 22% and 33%, resp. Dextran restores glomerular filtration and renal blood flow more rapidly to greater levels and for a longer period of time than oxypolygelatin. It also exhibits about twice the effect of withdrawing fluid from the interstitial compartment.

Toxicology of organic phosphorus-containing insecticides to mammals.

DU BOIS, K. P., AND COON, J. M. *Archi. Ind. Hyg., 6,* 9-13 (1952).

The org. phosphorus-contg. insecticides may be classified into three groups, namely, the alkyl pyrophosphates, the alkyl thiophosphates, and the phosphoramides. All of the members of these groups which are in wide current use as insecticides are highly toxic to insects and mammals. In the development of compds. with high insecticidal activity and low mammalian toxicity the most promising results thus far have been obtained in the thiophosphate series. All of the org. phosphorus-contg. insecticides appear to owe their principal pharmacological actions to their ability to inhibit cholinesterase, thus increasing the acetylcholine content of the tissues with resultant intensified activity of structures which are normally stimulated by acetylcholine. The pyrophosphates and the alkyl thiophosphates are active inhibitors of cholinesterase in vitro and inhibit the enzyme activity of mammalian and insect tissues in vivo. The phosphoramides differ from the other groups of phosphorus-contg. insecticides in exerting their effects mainly on peripheral tissues. In addn. they are stable toward hydrolysis and have a long duration of action.

The toxicity and urinary excretion of crystalline metaphosphates.

GOSSELIN, R. E. March 18, 1952 (UR-192); *Nuclear Sci. Abstracts, 6,* 360 (1952).

In terms of toxicity, hydrolysis, and excretion in rats and rabbits, cyclic polymers of sodium phosphate contrast with linear polymers like cryst. tripolyphosphate and glassy hexametaphosphate. After parenteral administration of the cyclic compds. (sodium trimeta- and tetrametaphosphate), over 90% of the dose is recovered in the urine in an unhydrolyzed state, but a small fraction appears to be broken into orthophosphate residues. In contrast only 10% of sodium tripolyphosphate and about 20% of hexametaphosphate escape hydrolysis in the rat. Calcium salts of these compds. are excreted more slowly, and in consequence hydrolytic destruction is somewhat greater. All hydrolyses observed in vivo are presumably enzymatic. Toxicity offers a second biol. distinction between the cyclic and the linear polymers of sodium phosphate. Although none has a high acute toxicity in the rat, the latter are considerably more toxic than the former. This difference is discussed in the light of differences in hydrolysis and excretion.

NUTRITION

Dietary standards in the United States.

MAYNARD, L. A. *Public Health Reports, 67,* 784-7 (1952).

The recommended dietary allowances formulated by The Food and Nutrition Board of the Natl. Research Council are presented in a table. The allowances are intended to represent "levels enough higher to cover substantially all of the individual variations in the requirements of normal people." The dietary allowances are classified according to age, sex, and levels of activity. Emergency food supplies are discussed.

(Continued on page 18 following technical papers)

* These Selected Abstracts are made available to FOOD TECHNOLOGY through the cooperation of Associate Editor H. A. Campbell and the General Foods Corporation of New York, N. Y. The abbreviations found in these abstracts are similar to those used by Chemical Abstracts.

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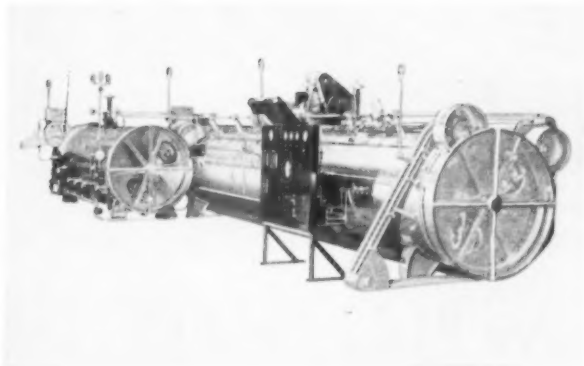

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A Simplified Procedure for Thermal Process Evaluation

M. PATASHNIK

Libby, McNeill & Libby Salmon Division Laboratory, Seattle, Washington

(Manuscript received July 20, 1952)

A simplified procedure for thermal process evaluation is presented. Process values attained during heat penetration tests may be readily evaluated immediately after each temperature reading.

The procedure involves taking container temperature readings at equal time intervals. Corresponding to each equal-time-interval temperature reading, lethal ratio (F/t) values are tabulated and totaled. The product of this sum and the equal-time-interval used gives the process value directly.

Lethal ratio tables for use with this proposed method are included.

The principal purpose in presenting this material is to give technicians and students lacking mathematical background a short, simple, understandable procedure for process evaluation. The procedure, to be delineated below, is similar to the "General Method" of Bigelow *(6)* and that of Ball *(4)* with the exception that no curves need be plotted once the slope of the thermal death time curve is known; the integration under the lethality curve may be accomplished by the simple addition of lethality values.

The problem of thermally processing canned foods involves 2 component factors. The first is bacteriological and is concerned with the thermal death times of micro-organisms which the thermal process is intended to destroy. More recent concepts as advanced by Ball *(5)* and Stumbo *(12, 13, 14)* speak in terms of "phantom" thermal death times or rates but these have a similar significance to the term thermal death time. The second is concerned with the physical factor of the time temperature relations within the can while it is being heated. We will demonstrate that these 2 factors can be very simply related.

Review of literature. The first systematic approach to thermal process evaluation was that of Bigelow, Bohart, Richardson and Ball in 1920 *(6)*. The basic concepts and methodology inherent in their original work are still in common use today. The authors set forth a graphical method of wide applicability, usually referred to as the "General Method," which combined bacteriologic data with heat penetration data.

In 1923 Ball *(2)* derived an analytic or "Formula Method" of evaluating processes, "in order to reduce the time necessary to obtain results, as well as to obtain a basis for coordinating the various factors which enter into the calculation." The mathematical formulations were accompanied by charts relating the variables which further simplified process evaluation. However, it was necessary to make a number of mathematical and empirical assumptions in order to facilitate and make possible a mathematical treatment of the subject. Therefore, under certain conditions results obtained by the "Formula Method" must be interpreted in the light of assumptions made in this work.

In 1928 Ball *(4)* followed up his original work with an extensive mathematical treatise dealing with the solution of actual problems on thermal processing of canned foods. This work was replete with problems, curves and tables intended for practical use, and embodied procedures for employing either the "Formula" or "General Method."

The ready application of Ball's methods to commercial food processing stimulated a vast amount of research which in turn resulted in a better understanding of the basic problems of thermal processing.

In attempting greater simplification of Ball's "Formula Method," Olson and Stevans in 1939 *(10)* evolved a "Nomographic Method" applicable to canned foods exhibiting straight-line semilogarithmic heating curves.

Improvements in the "General Method" for thermal process calculations were made in 1940 by Schultz and Olson *(11)* by the use of a special coordinate paper. Use of this paper simplified calculations and reduced the chance of misplotting points; but it had the practical disadvantage of requiring a different, specially printed coordinate paper for each new thermal-death-time slope or z value used in calculations. At the same time, they introduced formulae for converting heat penetration data from one initial food and/or retort temperature to another. These formulae apply to conduction and convection heating. Because they are very simple and rapid to use these formulae have greatly increased the applicability of the "General Method."

Despite the aforementioned improvements, process value calculations by the "General Method" are still relatively laborious.

Likewise process calculations by the simplified "Formula Method" of Ball still involves the time and effort of plotting the heat penetration data on semi-log arithmic paper to obtain its slope, f_h. We should also note that the "Formula Method" is directly applicable only in so far as actual test conditions are in reasonable accord with the assumptions involved in its derivation.

For an excellent treatment of the subject of thermal process evaluation, as well as the newer theoretical concepts, the reader is referred to Stumbo's review *(15)*.

Lethality tables and the definition of terms employed are included in the appendix.

PROPOSED SIMPLIFICATION OF GENERAL METHOD

Equal-time-interval procedure. If during heat penetration tests, temperature readings are taken at *equal time* intervals, process values can be accurately evaluated without plotting a curve. This is accomplished by tabulating the *lethality* value for each equal-time-interval *temperature* reading and making a summation thereof. The product of this sum and the equal time interval used gives the process value directly.

The example presented in Table 1 *(16)* below was selected for illustration of our procedure since it was already set up in the tabulated form we employ.

The equal-time interval procedure requires data as shown in Table 1, columns 1, 2 and 3. Column 1 represents time, and column 2 the container temperature data. Column 3 lists the

TABLE 1

Jar temperatures and lethal ratios during processing at 240° F. (115.6° C.) and cooling of green beans in a pint jar; process value calculations. Data from Toepfer et al. (16)

1	2	3	4	5
Time (Min.)	Temp. (° F.)	Lethal ratio (F/t)	Σ(F/t)	Process value, F' (Min.)
15	194	0.0008		
10	203	.0024		
5	211	.0068		
0[b]	222	.0278		
5	234	.1290		
10	237	.1900		
15	239	.2450		
20 steam cut	240	.2783	0.741	3.71 (F_H)
25	237	.1900		
30	236	.1670		
35	233	.1140		
40	228	.0600		
45	225	.0409		
50	222	.0278		
55	220	.0215		
60	211	.0068		
65	204	.0028		
70	194	.0008		
75	187	.0003		
80	181	.0001	1.512	7.56 (F_T)

[b] 0 indicates the beginning of processing time (time when retort reaches 240° F. (115.6° C.).

lethal ratios F/t corresponding to the temperatures of column 2, for $z = 18$.[a]

Our simple processing problem is now set up for solution: Based on the data in Table 1 what is the total process value, F_T, for the 20 minute process and 60 minute cool? The answer is found by adding all the values in column 3 which gives the value 1.512 shown in Column 4. This value when multiplied by the equal-time-interval of 5 minutes, gives a total process value $F_T = 7.56$ minutes shown in Column 5.

Considering the same data in Table 1, we may solve for the process values acquired during the heating and cooling phases of the process, F_H and F_C. Process value F_H is obtained by taking the summation of the values in column 3 through 0.2450 (15-minute time interval) to which is added ½ (0.2783) giving 0.741 shown in column 4. This when multiplied by the time of 5 minutes results in $F_H = 3.7$ minutes shown in column 5. The process value during the cooling p of the process ($FC = F_T - F_H$) is calculated to be $7.56 - 3.71 = 3.85$ minutes.

Basis of procedure. A plot of the lethal ratios F/t in column 3 against the time of heating in column 1 (Table 1) produces the lethality curve. This curve in effect combines bacteriologic time-temperature data and heat penetration time-temperature data under a single curve. Process value calculation whether by the "Formula" or "General Method" involves integration or area summation under this lethality curve. This area may be determined by counting squares, by measuring with a planimeter, or by the simple addition procedure we have employed if equal time intervals have been used. The equal time interval in essence is the reason why this simple addition procedure works.

Basically the integration is performed by employing the trapezoidal rule *(1)*—a rule used extensively by naval architects in evaluating areas under curvilinear figures. The rule states: "To find the area of a curvilinear figure . . . by means of the trapezoidal rule, divide the base into any convenient number of equal parts and erect perpendiculars to the base meeting the curve; then to ½ the sum of the first and last of these add the sum of all the intermediate ones; the result multiplied by the common distance apart will give the area required" *(1)*.

Since the first and last ordinates of a lethality curve are equal to zero the total area under the lethality curve (equal to the total process value F_T) may be obtained by summating the lethal ratio values F/t taken at equal time intervals and multiplying the resultant sum by the equal time interval.

[a] Note that Table 7 lists the F/t values for $z = 18$ to three places to the right of the decimal. Toepfer's *(16)* use of four places in Table 1 column 3 is unnecessary as the fourth place is insignificant.

The same principle may be employed to calculate the process value at *any* time interval of the process by a, adding all (F/t) values *before* the desired time interval, b, adding to this ½ the (F/t) value *at* the desired time interval and c, multiplying the summation resulting thereof by the equal time interval.

The accuracy of this equal interval integration procedure depends upon how closely a curvilinear area can be represented by its equivalent in equally spaced trapezoids, which depends on the size of the equal space interval employed. The choice of a suitable equal time interval in process evaluation offers little difficulty as will be shown.

EQUAL TIME INTERVAL PROCEDURE—SPECIAL CONSIDERATIONS

In the application of this integration method a number of questions may arise: a, What is the maximum equal time interval that should be used? b, What is the effect of sharp breaks, or sharply changing curvature in the lethality curve, upon calculations? c, To what extent are process value calculations affected when the peak of the lethality curve does not fall on an equal time interval?

We may answer the first question by stating that in our experimental work (50 to 95 minute salmon processing) we have found the 4- or 5-minute time interval both accurate and convenient. In the experience of the writer, the factors involved in the above questions have caused insignificant deviations, *well within* the experimental error or the inherent variations from can to can. To help us evaluate the quantitative process value differences that one might anticipate with *different* equal-time-intervals, we have solved some typical problems. The solution of these problems will throw some light on the above questions.

EFFECT OF VARYING THE "EQUAL-TIME-INTERVAL" UPON PROCESS VALUE CALCULATIONS

Consider the heat penetration data in column 1 and 2 (Table 2), based upon an actual commercial test of salmon, processed for 90 minutes at 242° F. (116.7° C.) in 301 x 411 cans (1 lb. tall).

The lethal ratios have been tabulated for $z = 21$. The lowest equal time interval of 2 minutes was intentionally chosen for demonstration purposes and was increased to 20 minutes in subsequent calculations. The process values F_H and F_T have been calculated as shown in column 5 for the 2-minute equal time interval.

In Table 3 we have arranged a summary of the calculated F_T and F_H values based on the data in Table 2 for time intervals of 2 to 20 minutes. In performing these calculations the equal time intervals were spaced off in both directions from the 90-minute time interval. Thus, for the 10-minute equal time interval, calculations were based on the starred (*) lethal ratios in Table 2. The percent deviation figures were expressed relative to the F_T and F_H values for the 2.0 minute interval.

Deviations for the 4- and 6-minute time interval were less than 1% and for the 8- to 20-minute time interval less than 5%.

Although calculations based upon equal time intervals as high as 20 minutes displayed deviations of less than 5.0%, in practice we have found the 4- or 5-minute equal time interval both rapid and accurate. Calculations based on such widely spaced time intervals as 8 to 20 minutes are still reasonably accurate because of the reversing curvature of the lethality curve. Thus, during the trapezoidal integration, gains in area above the lethality curve and losses in area below the curve nearly cancel one another. To afford a clearer insight into the physical approximations involved in each of the Table 3 calculations, one could plot the lethality curve from the 2-minute equal time interval data of Table 2. Then by connecting the F/t ordinates for each *set* of equal time intervals used in calculations by straight lines,

TABLE 2

Center can temperatures and lethal ratios during commercial processing of salmon for 90 minutes at 242° F. (116.7° C.) in 301 × 411 cans (1-lb. tall)

1 Time (Min.)	2 Temp. (° F.)	3 Lethal ratio (F_t/t)	4 $\Sigma(F_t/t)$	5 Process value, F' (Min.)
34	185	0.001		
36	189	.001		
38	193	.002		
40	197*	.003*		
42	201	.005		
44	204	.006		
46	207.5	.010		
48	210	.012		
50	213*	.017*		
52	215.5	.022		
54	217.5	.028		
56	219	.033		
58	221	.042		
60	223*	.052*		
62	225	.065		
64	226.5	.076		
66	228	.090		
68	229	.100		
70	230*	.112*		
72	231	.125		
74	232.5	.147		
76	233.5	.164		
78	234	.173		
80	234.5*	.183*		
82	235.5	.204		
84	235.8	.210		
86	236	.215		
88	236.5	.227		
90 steam cut	237*	.240*	2.445	4.89 (F_H)
92	237.5	0.254		
94	237.5	.254		
96	237.5	.254		
98	237	.240		
100	236*	.215*		
102	234	.173		
104	232	.139		
106	230	.112		
108	226.5	.076		
110	224*	.058*		
112	220.5	.039		
114	218	.030		
116	215	.021		
118	212	.015		
120	210*	.012*		
122	208	.010		
124	206	.008		
126	203.5	.006		
128	201	.005		
130	198*	.003*		
132	196.5	.003		
134	194	.002		
136	192.5	.002		
138	191.5	.002		
140	190*	.001*		
142	189	.001		
144	188	.001		
146	187	.001		
148	186	.001		
150	185*	.001	4.504	9.01 (F_T)

NOTE: Come up time = 10 minutes.
* Values used for 10 minute equal time interval calculation.

TABLE 3

The effect of increasing the equal time interval from 2 to 20 minutes upon calculated process values F_T and F_H. (Based on processing salmon 90 minutes at 242° F. (116.7° C.) as per Table 2 data)

Equal time interval (Min.)	Calculated process value F_H	Percent deviation from 2 min. calc.	Calculated process value F_T	Percent deviation from 2 min. calc.
2	4.89	0.0	9.01	0.0
4	4.92	+0.6	9.04	+0.3
6	4.90	+0.2	8.96	−0.6
8	5.01	+2.5	9.15	+1.6
10	4.87	−0.4	8.97	−0.4
12	4.99	+2.0	8.98	−0.3
14	5.06	+3.5	8.96	−0.6
16	5.02	+2.7	8.93	−0.9
18	4.93	+0.8	8.59	−4.7
20	4.98	+1.8	8.62	−4.3

the area of the approximated lethality curve may be compared to that for the 2-minute time interval curve.

In Table 2 the process steam was cut at 90 minutes, yet the peak temperature was reached and maintained *after* the cessation of steam flow. In this instance the rise was only 0.5° F. (.28° C.) but in other tests rises of 3° F. (1.67° C.) were noted. Since we generally locate an equal time interval at the end of the cook, the rise in temperature after this point *may* result in calculated process values (F_T) slightly on the low side, if too great a time-interval has been chosen. (Such rises in temperature are frequently the case when cooling is conducted outside the retort). The increasing negative F_T deviations in Table 3 are partly due to this cause.

We have employed equal time intervals as high as 20 minutes mainly to demonstrate that even where a substantial calculation error was anticipated, results were not too far off. However, by using considerably shorter time intervals we *know* that results will have a high degree of accuracy.

The above problem involved a rather slow heating product where heat transfer was largely by conduction.

Now let us consider a rapid heating product where heat transfer is primarily by convection. The data of Table 1 will serve for this purpose. An entire calculation in its simple form is shown in Table 4 for the 10-minute equal time interval. Table 5 summarizes the calculated F_T and F_H values for equal time intervals of 5 to 20 minutes.

TABLE 4

Process value calculation for 10-minute equal time interval. (Green beans processed 20 minutes at 240° F. (115.6° C.); based on Table 1 data)

1 Time (Min.)	2 Temp. (° F.)	3 Lethal ratio (F/t)	4 $\Sigma(F/t)$	5 Process value, F' (Min.)
10	203	0.002		
0	222	.028		
10	237	.190		
20 steam cut	240	.278	0.359	3.59 (F_H)
30	236	.167		
40	228	.060		
50	222	.028		
60	211	.007		
70	194	.001	0.761	7.61 (F_T)

TABLE 5

The effect upon calculated process values F_T and F_H, of increasing the equal time intervals from 5 to 20 minutes. Green beans processed 20 minutes at 240° F. (115.6° C.); based on Table 1 data)

Equal time interval (Min.)	Calculated process value F_H (Min.)	Percent deviation from 5 min. calc.	Calculated process value F_T (Min.)	Percent deviation from 5 min. calc.
5	3.71	0.0	7.56	0.0
10	3.59	−3.3	7.61	+0.7
15	4.05	+9.2	8.31	+9.9
20	3.34	−10.0	7.56	0.0

The 10-minute equal time interval calculation compares favorably with the 5-minute results. Its deviation for F_H and F_T was respectively −3.3% and +0.7%. Differences of such magnitude are generally well within the variations in bacteriological data and the physical variations between test containers for many food products. Thus, where the scope of an investigation is quite large and results accurate to within 5% are acceptable, it is possible to shorten calculations by employing more widely spaced equal-time intervals. However, for conventional process evaluation the 4- to 6-minute time interval is preferred. A shorter time interval might be necessary for small cans with convection heating products because they heat and cool very rapidly.

CALCULATION OF PROCESS VALUES DURING HEAT PENETRATION RUN

Sometimes it is desirable to know the process value *during* the heat penetration run. In certain tests it

would be highly advantageous to have such knowledge immediately after each equal-time-interval temperature reading.

When the process value at each equal time interval during the process is desired, calculations are carried out according to Table 6. With the aid of an adding or

TABLE 6

Procedure for calculation of process values at each equal time interval during the heat penetration test. (Data from Table 1)

1	2	3	4	5
Time (Min.)	Temp. (° F.)	Lethal ratio (F/t)	(F/t)	Process value, F' (Min.)
15	194	0.001	0.000	
10	203	.002	.002	0.01
5	211	.007	.006	0.03
0	222	.028	.024	0.12
5	234	.129	.102	0.51
10	247	.196	.262	1.31
15	259	.245	.479	2.40
20 steam cut	240	.278	.741	3.71
25	237	.190	.975	4.88
30	236	.167	1.153	5.77
35	233	.114	1.294	6.47
40	228	.060	1.381	6.91
45	225	.040	1.431	7.16
50	222	.028	1.465	7.33
55	220	.022	1.490	7.45
60	211	.007	1.504	7.52
65	204	.003	1.509	7.55
70	194	.001	1.512	7.56

calculating machine the tabulation is effortless. Prior to the start of the heat penetration test the equal-time-interval process times to be used are entered into column 1. A table of F/t values for the product being tested is on hand. As each temperature reading is recorded in column 2, the lethal ratio F/t is entered in column 3 from which the summation values of column 4 are derived by cumulative addition. The latter multiplied by the equal-time-interval gives the process values shown in column 5.

With our equipment *(7)* we usually run 4 to 6 test cans. It is possible for one man to run the entire heat penetration test and have complete calculations during the course of the test. Thus, process steam may be cut and cooling started at any desired process value, F_H. In practice, these calculations are carried out for the slowest or two slowest heating cans during the test. Calculations for the faster heating cans are completed within a few minutes after the completion of the heat penetration test.

A graphical representation of Table 6 data is shown in Figure 1. Also shown by the way of comparison is a similar curve for a slow heating product based on Table 2 data using a 4-minute equal-time-interval.

CONCLUSION

Our laboratory has made extensive use of the described process evaluation procedure. It has been used in routine checking of cannery retort practice, in developing new processes, and in studying the quality effects of both over and under processing. Processing studies were considerably expedited by employing the simplified method herein developed.

For the sake of brevity, we have intentionally avoided elaborating on the different types of processing problems that can be solved by the equal-time-interval method.

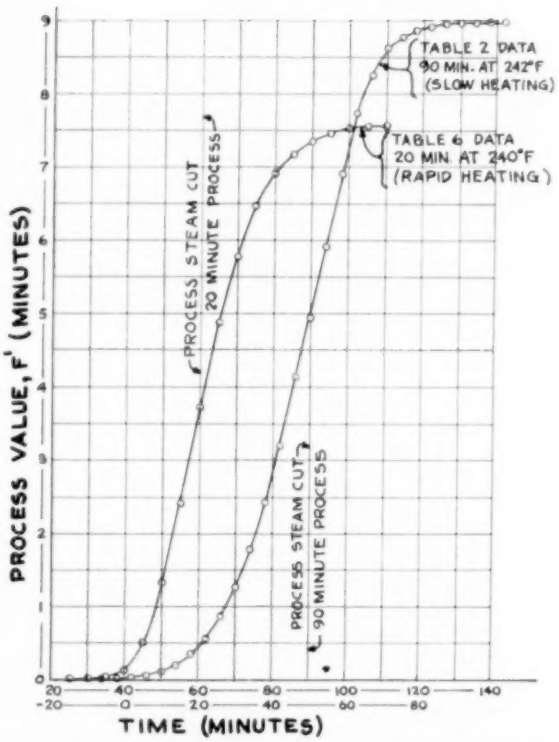

Figure 1. Showing increase of process value during heat penetration test.

However, it might be pointed out that process values (F_H or F_T) for shorter and somewhat longer process times than shown in Tables 2 or 6 can be evaluated in a few minutes by referring to the already tabulated values in these tables. Further a given set of heat penetration data for one retort and/or initial temperature may be readily transformed to another by the method of Schultz and Olson *(10)*. This gives greater flexibility to the proposed procedure. The reliability of such transformed data has been experimentally verified in our salmon processing studies.

In making studies similar to that of Fagerson and Esselen *(8)* on F_o distribution patterns for convection and conduction type of heating the use of the proposed procedure is suggested in lieu of the General (graphical) Method of Bigelow *et al. (6)*. Similarly, in making heat penetration temperature-distribution pattern studies similar to that of Jackson and Olson *(9)*, data taken at equal time intervals would be highly advantageous for ready conversion to F values.

In treating heat penetration data that have not been recorded at equal time intervals it is still possible to employ the equal-time-interval procedure. However, it would first be necessary to plot the time temperature data. From this plot, temperatures corresponding to the equal time intervals can be tabulated. The procedure from this point on would be the same. However, the main advantage of time saving would be lost.

Townsend *et al. (17)* in their comparative heat penetration studies on jars and cans took temperature readings "at ½- or 1-minute intervals during rapid heating

and 5-minute intervals during slow heating." Due to the extensive nature of this investigation, substantial savings in time would have been possible by employing the largest equal time interval consistent with the desired accuracy of results. In future investigations of this type it might be well to consider this point. The data in Table 3 and 5 can be used as a rough guide in this respect.

In employing the proposed procedure on salmon processing we have used a standard 10-minute come-up time. Process time was started at the end of this period. The process values thus obtained applied to this standard come-up time. For other come-up times the equivalent *process time* correction may be assumed to be 42% of the come-up time difference *(3, 4)*. In the absence of more accurate data, this roughly empirical assumption is generally employed. However, more closely to establish the effect of different come-up times upon *process time*, the equal-time-interval procedure itself may be used.

APPENDIX

Definition of Terms and Lethal Ratio Tables

F = Represents the time in minutes required to reduce a given number of organisms at 250° F. (121.1° C.) to some prescribed safe level.

z = Represents the slope of the thermal death time curve expressed as the abscissa interval in °F. intercepted by the curve in passing through one logarithmic cycle on semi-logarithmic paper.

F/t = The lethal ratio F/t at a given temperature is the ratio of the time in minutes (F) required to reduce a given number of organisms to some prescribed safe level at 250° F. (121.1° C.), to the time in minutes (t) to accomplish the same reduction at any other temperature (T). It is a measure of the relative lethal or killing effect upon an organism at any temperature expressed as a fraction of the effect at 250° F. (121.1° C.). Mathematically, $F/t = \frac{1}{\log^{-1}\frac{250 - T}{z}}$. Since the lethal ratio F/t is a function of z and T it may be tabulated for given z and T values. Table 7 (z = 18) and Table 8 (z = 21) have been prepared for actual use in process calculations up to 250° F. (121.1° C.). Table 9 (z = 17 to 23 inclusive) has been set up in the *general* form in which this table should be eventually developed for practical use. The temperature intervals should be closer and extended to higher temperatures. Also, the z values should cover the entire range of z values encountered in commercial practice. The substantial variation of lethal ratio (F/t) values with z in Table 9 emphasises the importance of using accurate z values in process evaluation.

F_o = Represents the F value of a process in terms of equivalent minutes at 250° F. (121.1° C.) for the assumed con-

TABLE 7

Table of lethal ratio (F/t) values for z = 18

T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio
191	0.001	219	0.019	233.5	0.121	243.25	0.422
192	0.001	220	0.022	234	0.129	243.50	0.436
193	0.001	220.5	0.023	234.5	0.138	243.75	0.450
194	0.001	221	0.025	235	0.147	244	0.464
195	0.001	221.5	0.026	235.5	0.157	244.25	0.479
196	0.001	222	0.028	236	0.167	244.50	0.495
197	0.001	222.5	0.030	236.5	0.178	244.75	0.511
198	0.001	223	0.032	237	0.190	245	0.527
199	0.001	223.5	0.034	237.5	0.202	245.25	0.545
200	0.002	224	0.036	238	0.215	245.50	0.563
201	0.002	224.5	0.038	238.5	0.230	245.75	0.581
202	0.002	225	0.041	239	0.245	246	0.600
203	0.002	225.5	0.044	239.25	0.253	246.25	0.620
204	0.003	226	0.047	239.50	0.261	246.50	0.640
205	0.003	226.5	0.050	239.75	0.269	246.75	0.660
206	0.004	227	0.053	240	0.278	247	0.681
207	0.004	227.5	0.056	240.25	0.287	247.25	0.704
208	0.005	228	0.060	240.50	0.296	247.50	0.727
209	0.005	228.5	0.064	240.75	0.305	247.75	0.750
210	0.006	229	0.068	241	0.315	248	0.774
211	0.007	229.5	0.072	241.25	0.326	248.25	0.800
212	0.008	230	0.077	241.50	0.337	248.50	0.826
213	0.009	230.5	0.082	241.75	0.348	248.75	0.853
214	0.010	231	0.088	242	0.359	249	0.880
215	0.011	231.5	0.094	242.25	0.371	249.25	0.910
216	0.013	232	0.100	242.50	0.383	249.50	0.940
217	0.015	232.5	0.107	242.75	0.395	249.75	0.970
218	0.017	233	0.114	243	0.408	250	1.00

TABLE 8

Table of lethal ratio (F/t) values for z = 21

T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio	T Temp. (°F.)	F/t Lethal Ratio
181	0.001	206	0.008	225.5	0.068	238	0.268
182	0.001	207	0.009	226	0.072	238.5	0.283
183	0.001	208	0.010	226.5	0.076	239	0.299
184	0.001	209	0.011	227	0.080	239.5	0.316
185	0.001	210	0.012	227.5	0.085	240	0.334
186	0.001	211	0.013	228	0.090	240.5	0.353
187	0.001	212	0.015	228.5	0.095	241	0.373
188	0.001	213	0.017	229	0.100	241.5	0.394
189	0.001	214	0.019	229.5	0.106	242	0.416
190	0.001	215	0.021	230	0.112	242.5	0.440
191	0.002	216	0.024	230.5	0.118	243	0.464
192	0.002	217	0.027	231	0.125	243.5	0.491
193	0.002	218	0.030	231.5	0.132	244	0.518
194	0.002	219	0.033	232	0.139	244.5	0.548
195	0.002	220	0.037	232.5	0.147	245	0.578
196	0.003	220.5	0.039	233	0.155	245.5	0.611
197	0.003	221	0.042	233.5	0.164	246	0.645
198	0.003	221.5	0.044	234	0.173	246.5	0.682
199	0.004	222	0.047	234.5	0.183	247	0.719
200	0.004	222.5	0.049	235	0.193	247.5	0.761
201	0.005	223	0.052	235.5	0.204	248	0.803
202	0.005	223.5	0.055	236	0.215	248.5	0.849
203	0.006	224	0.058	236.5	0.227	249	0.896
204	0.006	224.5	0.061	237	0.240	249.5	0.948
205	0.007	225	0.065	237.5	0.254	250	1.000

TABLE 9

Table of lethal ratio (F/t) values for z = 17 to 23

Temp. (°F.)	z Values						
	23	22	21	20	19	18	17
180	0.001	0.001	0.000	0.000	0.000	0.000	0.000
5	.001	.001	.001	.001	.000	.000	.000
190	.002	.002	.001	.001	.001	.000	.000
5	.004	.003	.002	.002	.001	.001	.001
200	.007	.005	.004	.003	.002	.002	.001
2	.008	.007	.005	.004	.003	.002	.002
4	.010	.008	.006	.005	.004	.003	.002
5	.011	.009	.007	.006	.004	.003	.002
6	.012	.010	.008	.006	.005	.004	.003
8	.015	.012	.010	.008	.006	.005	.003
210	.018	.015	.012	.010	.008	.006	.004
2	.022	.019	.016	.013	.010	.008	.006
4	.027	.023	.019	.016	.013	.010	.008
5	.030	.026	.022	.018	.014	.011	.009
6	.033	.029	.024	.020	.016	.013	.010
8	.041	.035	.030	.025	.021	.017	.013
220	.050	.043	.037	.032	.026	.022	.017
2	.061	.053	.046	.040	.034	.028	.023
4	.074	.066	.058	.050	.043	.036	.030
5	.082	.071	.065	.056	.047	.041	.034
6	.090	.081	.072	.063	.055	.046	.039
8	.111	.100	.090	.079	.069	.060	.051
230	.135	.123	.112	.100	.089	.077	.067
2	.162	.152	.139	.126	.113	.100	.087
4	.201	.187	.173	.159	.144	.129	.114
5	.222	.208	.193	.178	.162	.147	.131
6	.246	.231	.215	.200	.183	.167	.150
8	.301	.285	.268	.251	.233	.215	.197
240	.369	.351	.334	.316	.298	.278	.258
2	.448	.433	.416	.398	.379	.359	.339
4	.548	.534	.517	.501	.484	.464	.444
5	.606	.592	.573	.562	.546	.527	.508
6	.670	.658	.645	.631	.615	.599	.582
8	.818	.811	.803	.794	.785	.774	.762
250	1.000	1.000	1.000	1.000	1.000	1.000	1.000
2	1.221	1.233	1.245	1.260	1.273	1.292	1.312
4	1.493	1.520	1.550	1.585	1.623	1.668	1.720
5	1.650	1.687	1.730	1.777	1.833	1.895	1.966
6	1.823	1.874	1.930	1.994	2.070	2.154	2.254
8	2.226	2.310	2.404	2.511	2.635	2.783	2.955
260	2.722	2.848	2.993	3.163	3.414	3.594	3.871

dition $z = 18$. F_s serves as a commercial standard whereby the sterilizing values of different processes may be compared for this special condition *(4)*.

F_T = Total process value, representing the number of equivalent minutes at 250° F. (121.1° C.) contributed toward organism destruction, during the heating plus cooling phases of the process, for the z value used.

F_H = Process value representing the number of equivalent minutes at 250° F. (121.1° C.) contributed toward organism destruction, during the *heating* phase of the process, for the z value used.

$F_C = F_T - F_H$ = Process value representing the number of equivalent minutes at 250° F. (121.1° C.) contributed toward organism destruction during the *cooling* phase of the process, for the z value used.

F' = Process value representing the number of equivalent minutes at 250° F. (121.1° C.) contributed toward organism destruction at *any* time after the start of the process, for the z value used.

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Investigations Relating to the Possible Use of Atomic Fission Products for Food Sterilization[a,b]

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(Manuscript received July 28, 1952)

A coordinated research program of wide scope has been initiated by the United States Atomic Energy Commission, directed toward the harnessing of atomic energy for peacetime uses in the food industries. A collective progress report is given regarding the projects being conducted, as part of this program, with the food technology facilities of a number of outstanding educational institutions.

The materials known as fission products are isotopes (within the atomic weight range of 62 to 162) that result from the splitting of Uranium-235 by neutron absorption. These isotopes or fission products retain a portion of the energy of fission and release this energy by radioactive decay. Since 1944 the operations of the U. S. Atomic Energy Commission have been producing large quantities of radioactive fission products as by-products of the operation of nuclear reactors or atomic piles. The use of new reactors will amplify the supply. It is expected that these fission products will become available in considerable volume in the future, although up to now they have been stored in underground tanks as part of the war-born expediency and until adequate technology for processing could be developed.

Originally it was planned to limit research to securing basic knowledge in the technique of handling these sources of radioactive power and to investigate their fundamental effects in physical and chemical reactions, before industrial applications of these sources should be attempted. However, the active interest of various industries has indicated an immediate need of

[a] Contribution No. 139 from the Department of Food Technology, Massachusetts Institute of Technology.

[b] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 11, 1952.

learning what practical applications may be feasible, and research for basic knowledge has, therefore, been combined with research on industrial applications.

To this end, a coordinated research program of wide scope has been initiated by the Atomic Energy Commission, directed toward the harnessing of atomic energy for peacetime uses in the food industries. In the forefront of this program is the possibility that, for the sterilization of foods *(4, 7, 13-15, 18, 19)*, methods may be evolved that utilize the waste products available from the manufacture of atomic bombs. Atomic fission by-products may be made available for this purpose sometime in the future, if research indicates the feasibility of using such by-products *(8, 9)*.

RESEARCH FACILITIES

In this program, which is being pursued with vigor along many lines, the food technology research facilities of a number of outstanding educational institutions are being used. In other institutions, chemical and engineering projects are underway. Included among these institutions are the following: Brookhaven National Laboratory, Columbia University, Massachusetts Institute of Technology, Rensselaer Polytechnic Institute, Schering Corporation, Stanford Research Institute, University of Chicago, University of Michigan, Vitro Corporation, and Yale University.

Stanford Research Institute. In the original planning of this program, the need was recognized of securing advice from the food processing and similar allied industrial industries regarding their various problems and likely areas of feasible research. A techno-economic survey was, therefore, made for the Atomic Energy Commission by a team of investigators at the Stanford Research Institute in California, with the object of obtaining a cross-section sample of the opinions of representative technical groups in industry *(20)*.

The report of the findings of the Stanford Research Institute suggested that a potential large-scale industrial demand may exist for radioactive fission products. Extensive development of this market, however, will necessarily depend on favorable answers not now available and will be delayed for a few years while solutions are worked out to the many technical and economic problems known to exist. The report also indicated that perhaps the largest single potential demand for a confined radiation source is in the field of cold sterilization, specifically in drug sterilization and in food processing. This report recognized that long, arduous, and expensive research would have to be conducted to solve the many technical and management problems, and it encouraged the Atomic Energy Commission to seek solutions to these problems with the help of industry.

The following outline indicates the scope and general nature of the various projects that have been initiated to date or are contemplated when radioactive sources are available.

Brookhaven National Laboratory. The Brookhaven National Laboratory has been established as a central overall coordinating facility *(11, 12)* in the research and development program, with primary functions as follows:

(a) To advise the AEC in evaluation of research proposals and to aid operating contractors and industry in the establishment of safe and operable experimentation with high-level radioactive sources.

(b) To provide high-level (kilocurie) sources of isotopes irradiated in the Brookhaven National Laboratory reactor, to serve temporarily as mock sources preliminary to the availability of similar fission product sources.

(c) To provide an irradiation service at the Brookhaven National Laboratory for industry, utilizing kilocurie sources of gamma and beta emitters such as Cobalt-60, Tantalum-182, and Palladium-109. This service has been used to date for experiments both by food companies and pharmaceutical manufacturers.

(d) To stimulate and foster interest as well as advance the state of knowledge in certain experimental projects.

Columbia University. At Columbia University, a project is under way to study the possibility of sterilization of milk by Cobalt-60 radiation as well as to sterilize sufficient quantities of milk for animal feed studies *(5)*. The reason for feeding studies is to determine whether any toxic substance is produced by irradiation with Cobalt-60.

Other studies *(6, 10)* relating to chemical engineering applications are also under way at Columbia University, in which the same source is being used.

General Electric Company. A project has been started at the General Engineering Laboratory of the General Electric Company having various objectives relating to the industrial utilization of fission products *(3)*. A work program consisting of 3 phases has been established:

1. Feasibility study (to be supplied largely by the report of the techno-economic study by Stanford Research Institute).
2. Fission products source development.
3. Direct demonstration of the application of fission products sources for such purposes as:
   - (a) Sterilization of foods and drugs
   - (b) Industrial radiography
   - (c) Instrumentation

Massachusetts Institute of Technology. The research at the Massachusetts Institute of Technology may be cited as an example of a project selected by reason of more intimate knowledge concerning it on the part of one author. In the Department of Food Technology at this Institute, a program has been initiated on studying the effects of high-energy gamma rays from a high intensity Cobalt-60 source *(16, 17)*. The overall program is designed to study the many factors involved in arriving at a decision regarding the feasibility and practicability of sterilization of foods, drugs, and pharmaceuticals by isotopic radiations.

Studies are also under way at the Massachusetts Institute of Technology to determine the feasibility of using isotopic (gamma) radiations for eradication of insects in grain and flour. Previous studies with the use of cathode rays have shown this to be entirely practical, because low doses are required for insects. As millions of dollars worth of grain are lost annually in the

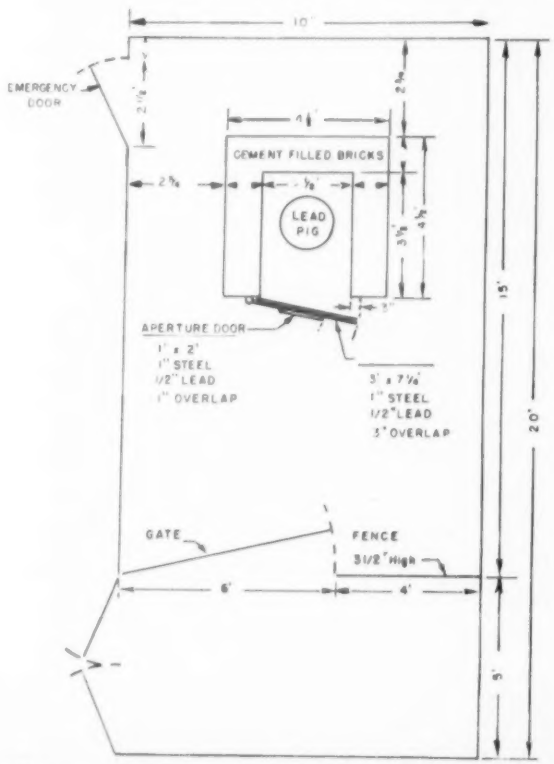
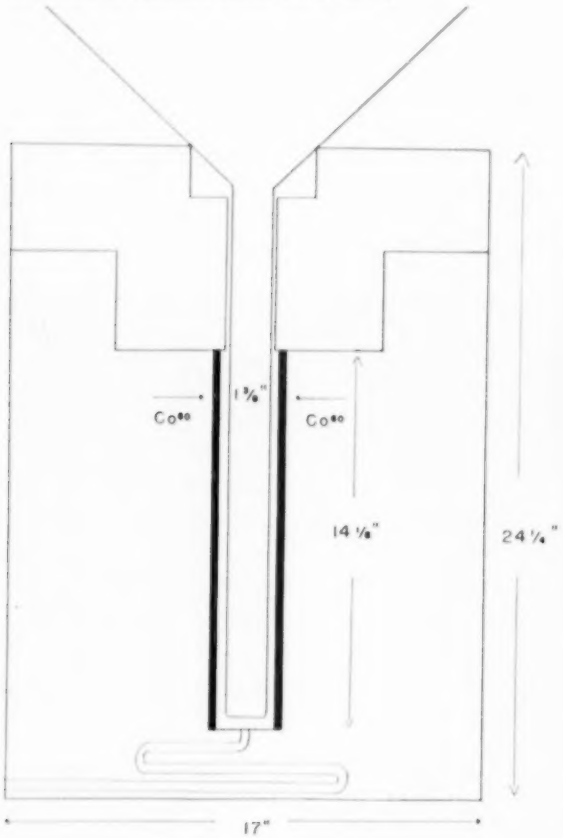


Figure 1. Plan view of food irradiation building at the Massachusetts Institute of Technology.

United States because of insect infestation, this problem is of great economic importance.

To this end a kilocurie Cobalt-60 source has been installed in a building especially designed and constructed for this purpose at MIT. (See Figure 1.) A diagrammatic sketch of the Cobalt-60 container is presented in Figure 2 and a floor plan of the Cobalt-60 vault and building in Figure 3.



COBALT-60 CONTAINER

Figure 2. Diagrammatic sketch of Cobalt-60 container at MIT.

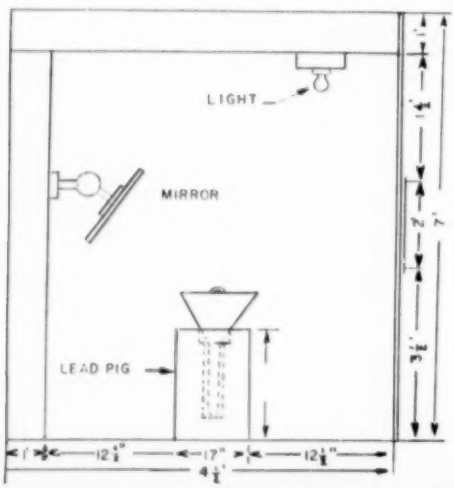


Figure 3. Side elevation of Cobalt-60 vault at MIT.

The operation of the source at MIT is illustrated in Figures 4 to 7. Even with the small aperture door of the vault open, the total radiation 3 feet from the door, at the wooden barrier, is less than 2 milliroentgens per hour. Hence, even with the aperture door open, one could work without radiation hazard for more than 8 hours per day. As an added safety precaution, however, the door is closed except when samples are inserted or removed from the pig.

At MIT the source has been calibrated by 2 chemical technics, each independent of the other. Calibration shows that the source has a radiation flux at the center of 2,000 rep per minute.

Research proceeding there at present includes comparative studies on the effects of radiations from the Cobalt-60 source and radiations from both cathode rays and X-rays produced by particle accelerators, on microorganisms and biochemical compounds. Such comparative studies are necessary to correlate isotopic sterilization efficiency with the efficiency data for other sources of radiation reported in the existing literature in the field, which deals predominantly with radiations from particle accelerators.

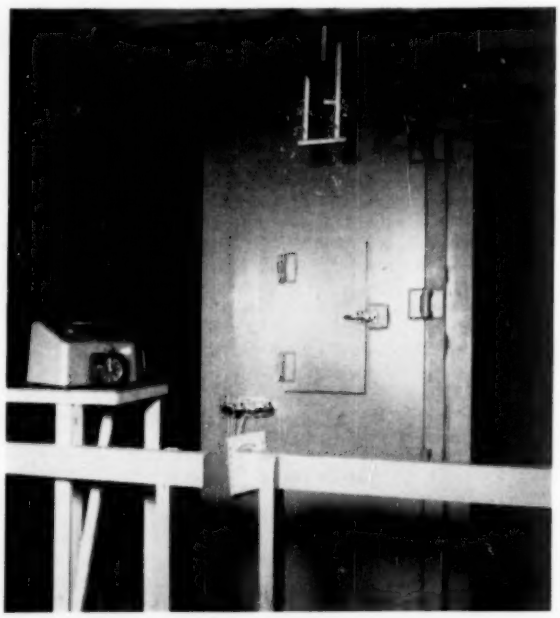


Figure 4. The MIT Cobalt-60 vault, door closed.

University of Michigan. The University of Michigan has been authorized to establish a research and development program in selected projects of chemical and physical reactions, based on previous investigations performed by the University faculty in these fields. One project is the investigation of those chemical reactions that give promise of being catalyzed by the radiation from fission products. It is well known that electromagnetic radiation can interact with molecules and the atoms within molecules of matter and result in free ions, chemical radicals, or activated molecules.

At the University of Michigan also a comparison project is under way on the effect of radiations on foods. This project has been in progress since last year, and the results accumulated to date have been reported in 2 progress reports to the Atomic Energy Commission *(1, 2)*. Studies have also been made by the Michigan group on the lethal doses of Cobalt-60 radiations for various species of bacteria.

Rensselaer Polytechnic Institute and University of Chicago. Plans are in progress for the installation and use of mock fission sources at Rensselaer Polytechnic Institute and the University of Chicago. The source at the latter institution is to be used cooperatively by the Meat Research Foundation and the Food Research Institute.

Figure 5. The MIT Cobalt-60 vault, door opened, funnel in place.

Atomic Energy Commission. The New York Operations Office, Division of Technical Advisors, of the Atomic Energy Commission has had a major role in coordinating this program and has acted in an advisory and administrative capacity for a number of contractors.

SUMMARY

It is hoped that when a satisfactory method is developed for the sterilization of foods by waste products of atomic energy, this method may prove applicable for the sterilizing treatment also of antibiotics, pharmaceuticals, and tissues. Of extreme interest is the feasibility of sterilizing the antibiotics, which are heat sensitive and difficult to keep aseptic.

Even if and when the feasibility of large-scale industrial sterilization of foods and drugs by isotopic radiations is established, a number of problems will remain to be solved. Among these are the following:

(a) Possible concentration of fission products to obtain the greatest amount of radiation in as small a volume as possible.

(b) Decisions as to the use of gross or mixed fission products; beta vs. gamma emitters, or both.

(c) How to obtain the most efficient utilization of the radiations with a maximum of safety.

(d) The logistics for the provision of replacement sources for depleted sources.

Figure 7. Inserting funnel into lead pig in MIT food irradiation building.

Figure 6. Removing plug from lead pig in MIT food irradiation building.

(e) The engineering of an industrial-scale isotopic sterilization plant, which would of necessity include the problem of adequate shielding.

(f) The establishment of a price structure for fission products, which will assist in the development of significant industrial uses. The value-in-use of any developed application will be a considered factor.

(g) Development of the technology for obtaining these fission product sources in the form and at radiation levels required by any worthy application.

It should be apparent from this brief review that the information available to date is fragmentary and, until much more research has been done, any decisions concerning the practical commercial applications of fission by-products must be held in abeyance for lack of adequate information on which to base valid decisions.

It is predicted that these decisions cannot be made for some time to come. However, in view of the enthusiastic attack being made on several fronts by numerous quali-

fied research groups, many of the answers will be produced by the cooperative endeavors of the food technologists of the universities and food industries in the United States—cooperative endeavors that have been catalyzed by the Atomic Energy Commission and its technical staff.

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The Research Laboratory—Its Role in the Control of Food Products[a]

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Effective technological control of a product is contingent upon the ability to determine the physical and chemical nature of the finished product at will. This kind of control requires knowledge of the raw materials, the process and the product to an extent that can be obtained only through research. The role of research in control is examined by a schematic analysis of basic control operations and by the study of the various steps in the manufacture of coffee. It is concluded that although the primary objective of a research organization is usually process and product development and basic or long-range research, the varied training of research personnel and the specialized equipment available in a research laboratory are such that a significant proportion of research effort can be very profitably devoted to problems of control.

Many people have the impression that control laboratories and research laboratories have distinctly different objectives and functions. Although the idea of strictly separate objectives and functions may have had some basis in the past, it certainly has no basis with respect to research and control in the food industry at present.

Perhaps the usual distinction made between research and control laboratories is more apparent than real, the difficulty arising from our definition of the word "control." As so often happens, a simple word in common usage has a tendency to pick up a variety of meanings and associations. Without entering into a discussion of semantics, we should be able to agree that in technology to have effective control of a product means to have the power to determine the physical and chemical nature of the finished product at will. Since the attributes of the finished product depend upon the process by which it is made and the raw materials that enter into its manufacture, it follows that control of the finished product is tantamount to control of the process and raw materials. It is clear, of course, that control in this sense requires a great deal of knowledge—much more knowledge than any of us have. It is equally clear, also, that research is the means of obtaining the knowledge required to maintain or improve our control of a product.

Although the thesis that research leads to knowledge and knowledge gives power to control may appear to be self-evident, further elaboration may be justified because, in the words of the late Chief Justice Holmes, "It is sometimes more important to emphasize the obvious than to elucidate the obscure."

A further appreciation of the relationship between research and control may be gained by considering a hypothetical food manufacturer who has gained com-

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 10, 1952.

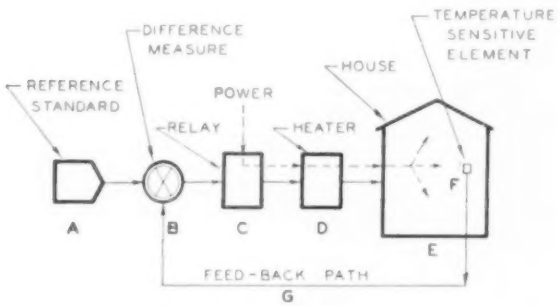
plete control of his process and product. This manufacturer would require no research facilities; the most elementary analytical "control" laboratory would suffice because, having complete manufacturing control of the situation at all times, he would be able to: automatically *modify his procedures* to compensate for any and all changes in his raw materials; smoothly *introduce substitute raw materials* into his procedures; *maintain maximum efficiency* in all operations at all times; *obtain "theoretical" yields* in every step; and *modify the flavor and texture of his product* at a moment's notice in accord with the latest disclosure of consumer preference.

You will perhaps agree that the above-described manufacturer is purely imaginary and that there is no doubt that research has an important role to play in the practical control of food products.

For the purpose of the present discussion, the role of research in control has been examined in 2 ways: first, by the schematic analysis of basic control operations and second, by a study of the various steps in the manufacture of an important food product (coffee) from the raw material to the finished product.

CONTROL SYSTEMS

Instrumental. Perhaps the most familiar automatic control system is the house heating system wherein a furnace is controlled by a wall thermostat. The elements of a system of this sort are shown in Figure 1. The diagram is not intended to be a



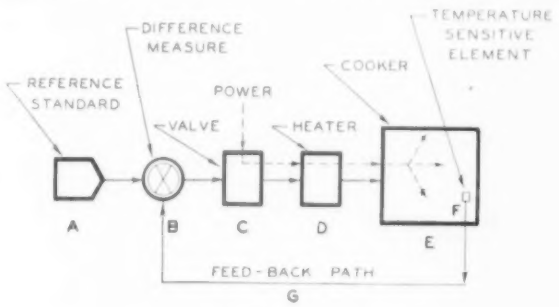
AUTOMATIC TEMPERATURE CONTROL SYSTEM
Figure 1

wiring diagram but represents, rather, the interrelation of the functional parts of the system. The combination of temperature sensitive element (F), reference standard (A) and difference measuring device (B) constitutes the thermostat. When the difference measuring device (B) indicates that there is a difference between (A) and (F), the relay (C) allows power and fuel to flow to the heater (D) whence heat is transmitted to the house (E). The temperature of the house is constantly compared with the reference standard by means of the difference measuring device. At a predetermined temperature, the relay cuts off the flow of power and fuel to the heater. The path (G) between (F) and (B) is called the feedback path for obvious reasons.

Although many instrumental controls are far more complex than the simple system illustrated here, the fundamental elements are always the same. The four elements common to all systems of instrumental control as shown in Figure 1 are:

(1) The reference standard, (A)
(2) The difference measuring device, (B)
(3) The relay or amplifier, (C) and
(4) The feedback path, (G).

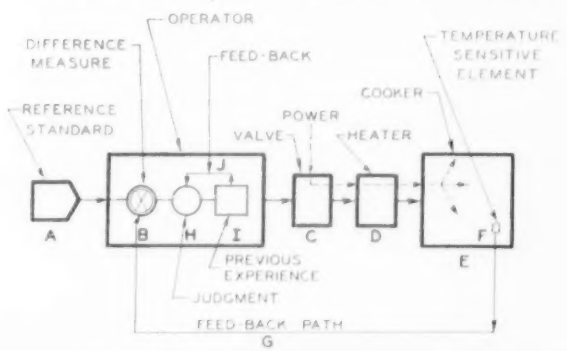
Manual control. The essential elements of a manually controlled system are not very different from those of an instrumentally controlled system. In Figure 2, a manually controlled cooker operation has been analyzed schematically. The analogy to the house heating system is apparent. To control the temperature of the cooker (E), the operator compares the temperature (F) of the cooker with a predetermined reference



MANUALLY CONTROLLED COOKER OPERATION
Figure 2

standard (A) and turns the valve (C) on or off depending upon the result of his comparison. The thermometer provides the information that is communicated along the feedback path (G). The operator's perception constitutes the difference measuring device (B).

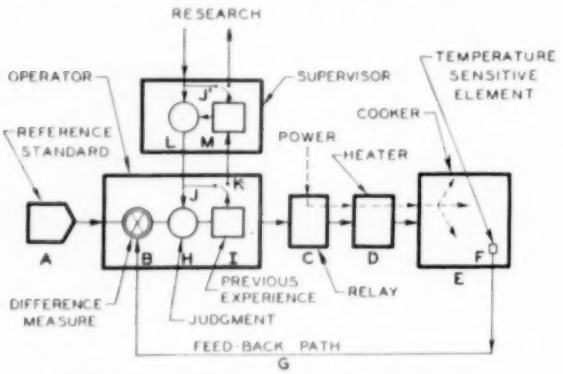
As may be expected if the operator can turn the valve only on or off, the temperature in the cooker will not be kept constant. A throttling control would be much better so that the valve could be adjusted in proportion to the imbalance of the system. There are, of course, instruments that do this, but for our present purpose, we are limiting ourselves to a manual operation. To achieve proper control, the operator must use his judgment in deciding how to adjust the control valve. The operator must, therefore, use both perception and judgment, separate functions which have been incorporated in the diagram of Figure 3. Here the symbols H and I have been added. H represents the operator's judgment, and I represents his previous experience with cookers. The difference perceived at B is transferred to H and then to I in a mental process with the feedback path J. By means of the circuit H, I, J, the operator visualizes the probable effect of a series of adjustments in the valve and recalls the degree of response produced in the system by similar changes encountered in his experience. On the basis of this combination of mental process, perception, judgment, and memory, the operator makes his decision, adjusts the valve, and in due course, observes the result of his action via the standard feedback path. This system, then, is potentially adequate to handle any operation and is limited only by the judgment and experience of the



MANUALLY CONTROLLED COOKER OPERATION
[THROTTLING CONTROL]
Figure 3

operator. As might be expected, his judgment and experience are rarely adequate for indefinite trouble-free operation in actual practice, and consequently, the system diagrammed in this slide is incomplete.

Process control. If the operator of a process is unable to accomplish his objective, he calls on his supervisor or superior who, on the basis of greater experience and more skilled judgment, can usually help the operator to get the process back on the track. This situation is expressed diagrammatically in Figure 4. Here a switch (K) is incorporated by means of which



PROCESS CONTROL THROUGH RESEARCH

Figure 4

the operator can engage the judgment (L) and experience (M) of his supervisor so that these will augment his own and enable him to regain control of the process.

This diagram also includes a switch that symbolizes the supervisor's option of referring an obstinate problem to research. The contribution of research is based on its function of combining experiment and observation with judgment and experience. In other words, the research organization can make experimental adjustments in a laboratory replica of the process, observe the results and make further changes as desired until the required information is obtained to be fed back to the supervisor and the operator.

Up to this point, a simple cooker operation has been used for the analysis of operations, but it does not require any great stretch of the imagination to visualize a manufacturing operation analyzed in the same fashion. The difference measuring device (B) in the diagram might correspond to a chemical or physical analysis applied to a food product in any stage of its manufacture. The reference standard (A) may constitute the specification which has been established by the management for the particular product. The relay (C) is any one of a variety of control points, and the feedback path (G) is the sample collection system.

THE BASIC ELEMENTS OF CONTROL

The 4 elements common to all systems of instrumental control are, therefore, also common to any kind of process control. These are the reference standard, the difference measuring device, the relay or control points, and the feedback path. Research has played a role in the practical development of each of these elements.

Reference standards. The reference standard takes many forms; it may be a written specification, it may be an analytical composition, it may be an approved sample of previous production, it may be a competitor's sample or it may be a sample of an experimental product produced in the laboratory or pilot plant. The selection of a reference standard or the development of specifications is often the objective of a research project in itself. A specification may be the result of a systematic analysis of a product over a period of time, or it may be derived from an analysis of consumer preference. Of course, the exact nature of the reference product is usually established by top management, but once established it is the function of the technologist to select objective measures of quality to serve as standards of comparison.

The difference measure. The difference measure also takes many forms. It may be a chemical or physical analysis, visual comparison, or taste panel evaluation. The development of these comparisons or methods of analysis is often a vital step in the gaining of control over a process. It is no exaggeration to say that one of the most sought after tests is one that is simple to perform, that is accurate and that reliably reflects variations in the product, thus enabling the prediction of final quality, shelf life, and general product performance in the hands of the consumer. The development of comparison methods, therefore, is a very important contribution of research to control.

The relay or control point. The control points in a process also cover a wide range. The first place control can be exerted is in the procurement or selection of raw materials. The next is in the formulation or blending of the materials, either raw materials or partially processed. The next point is in the processing. Here the control points are manifold: Time, temperature, the various machine settings, and equipment design are, in effect, control points. Finally packaging offers several control points. The method of packaging, the materials used, and the conditions of product storage, all may be involved. The contribution of research to all these elements of control needs no elaboration.

Feedback paths. The final element among the basic factors involved in process control is the feedback path. This is principally a scientific sampling step which may be carried out in the plant, the warehouse, or the field. Although not as prominent as other research activities, considerable research effort has been devoted to the development of methods of sampling.

RESEARCH AND CONTROL IN COFFEE MANUFACTURING

Examples of the role of research in control may readily be found by considering a single manufacturing operation from beginning to end. The manufacture of roasted coffee provides all of the desired examples.

THE MANUFACTURING OPERATION

Curing. The first step in the processing of coffee is the conversion of coffee cherries to green coffee by an operation known as curing. One of the best methods of curing coffee is the washed coffee process illustrated in Figure 5.

Briefly, in this process ripe red cherries are picked, and the skin and fibrous pulp are removed mechanically. The mucilage layer is digested by fermentation, then removed by washing, leaving a clean pale yellow parchment surface. The washed coffee in parchment is dried and the parchment removed by hulling machines to produce the characteristic green beans ready for export.

The quality of the green coffee produced in any given region is unpredictable. Consequently, the supply of quality coffee is often uncertain. The conclusion of our extensive research investigation, reported in part before the 1951 meeting of the Institute,[5] was that poor curing is responsible for much of the

[5] Johnston, W. R., and Foote, H. E. "Development of a New Process for Curing Coffee." *Food Technology*, **5**, 464 (1951).

low grade green coffee that is produced in many regions. Evidence was obtained that potentially good coffee can be damaged by unfavorable conditions during the fermentation and during the drying. As ordinarily practiced, the fermentation step is a spontaneous uncontrolled process brought about by a variety of microorganisms, and as a consequence, sour, off-flavors are often introduced into the coffee. The drying of the coffee is subject to the vagaries of the weather, and sometimes foul or musty flavors develop during the two weeks or more required to dry washed coffee on the patio. The discovery that coffee mucilage can be digested by a pectic enzyme preparation and that rapid mechanical drying of the washed beans is feasible has made possible a far greater control of the curing process. By the addition of pectic enzymes, the time required for mucilage digestion in the so-called fermentation step can be reduced to a few hours as compared to the 24 to 48 hours frequently required

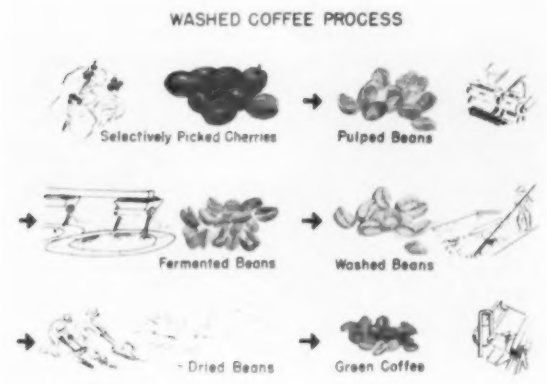


Figure 5

by spontaneous fermentations. Similarly with a modern mechanical dryer, the drying time can be reduced to a few hours. As a result of the improved control of curing, it is now possible to produce regularly, in any region, a coffee of uniform quality equal to the best coffee characteristic of the particular area. The value of this research to coffee curing is clearly based upon its contribution to the control of the process.

The coffee curing operation may be analyzed as follows:

Reference Standard	Standard [desired quality] sample
Difference Measure	Expert coffee tasters
Control Points	Mucilage digestion and drying
Feedback Path	Sampling procedure

Blending. The succeeding steps in the processing of green coffee are shown in Figure 6. The first step is the controlled formulation of the blend. This is accomplished by expert coffee tasters who, having established a suitable standard blend, must then select lots of green coffee that will reproduce the standard. Because of the many kinds of green coffee that are required to make a good blend and because of the variations in quality that occur, the formulation of the blend is a continuing process.

The coffee blending operation may be analyzed as follows:

Reference Standard	Standard blend sample
Difference Measure	Expert coffee tasters
Control Point	Formulation
Feedback Path	Sampling procedure

The role of research in the control of the coffee blending operation is relatively minor unless one considers the consumer research that may enter into the selection and adoption of the blend to be used as a reference standard.

Roasting. The roasting operation, that follows blending, requires careful control. This is the first operation in the series that is amenable to an objective method of measurement. Using a reflectance meter, shown in Figure 7, it has been possible to establish a numerical scale that corresponds to the degree of roast.

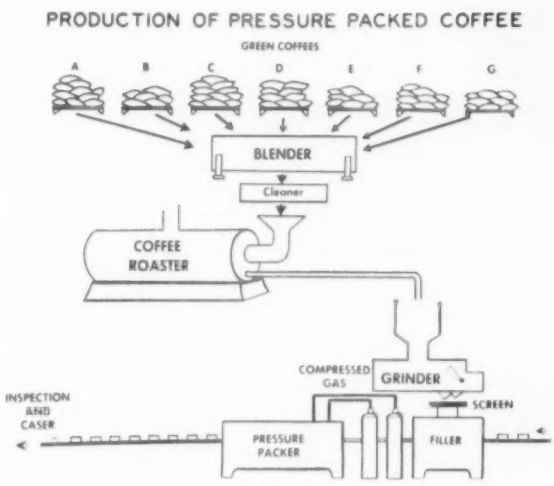


Figure 6

The coffee roasting operation may be analyzed as follows:

Reference Standard	Specified roast color
Difference Measure	Reflectance meter
Control Points	Time and temperature of roast
Feedback Path	Sampling procedure

This analysis places the reflectance meter as the difference measuring device, and the specified roast color number as the reference standard. The development of the physical method for determining roast color is the contribution of research to objective control of the roasting operation.

Grinding. Grinding is another process in which objective measurements enter into the control of the operation. The usual sieve analysis illustrated in Figure 8 is the difference measuring device in this case. Several different grinds are required for the various types of coffee makers now in vogue, and each grind has its specified standard sieve analysis. Determination of the best distribution of particle sizes [or sieve fractions] for any given purpose was the objective of one of our research projects.

The grinding operation may be analyzed as follows:

Reference Standard	Specified grind index
Difference Measure	Sieve analysis
Control Point	Mill setting
Feedback Path	Sampling procedure

The schematic analysis conforms to the same general pattern as the other operations that have been discussed.

Figure 7

Figure 8

Packaging. The final stage in the processing of coffee is packaging. The maintenance of roaster fresh flavor over long periods in transit and on the shelf has undoubtedly been the objective of a greater research effort than all other phases of regular coffee manufacture. The development of the pressure pack is an additional step in the same direction. In pressure packing, the freshly roasted ground coffee is placed in a special can; the air is removed by evacuation, replaced by oxygen-free nitrogen or carbon dioxide, and the can is then sealed at slightly above atmospheric pressure. The coffee particles immediately give off some of the gas contained in the cellular structure and a pressure of approximately ten pounds per square inch gradually builds up. The development of this pressure depends in part upon the promptness with which the coffee is moved from roaster to can. The pressure is measured with a manometric device as shown in Figure 9.

The packaging operation may be analyzed as follows:

Reference Standard ..... Pressure specification
Difference Measure ..... Pressure meter
Control Points—Speed of packaging, pressure at sealing and degree of roast
Feedback Path ..... Sampling procedure

It may be of interest to note that the consumer may play a role in the control system of this final stage of processing. Consumers are instructed to reject any can that does not show internal pressure when the top is pressed.

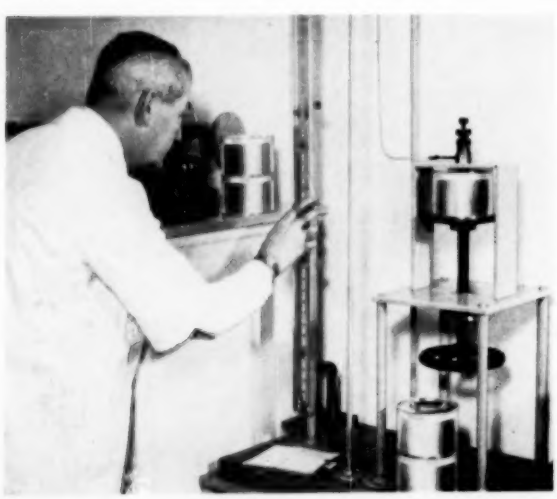


Figure 9

CONCLUSION

It is hoped that the foregoing analysis demonstrates the important role that research can and often does play in the control of food products.

To many people this picture of the relationship of research and control is not new, but some may have a reaction like that of the chap who heard a lecture on English Literature. He heard much of the wonders of poetry and prose. He was familiar with poetry, but prose was something new. After the lecture, he questioned the speaker. On receiving an explanation, he exclaimed with delight, "Well, what do you know? I, myself, have been writing prose all the time and I never knew it."

The central theme of this symposium is Research Planning. It should be in order, therefore, to conclude this discussion with a few suggestions for promoting fuller utilization of research in control. The research laboratory is usually established for new product development and basic or long-range research. Yet, because it has personnel with varied training and experience plus specialized equipment, the research laboratory is well suited to conduct investigations necessary for improved product control that may be difficult or impossible to pursue in the control laboratory. For this reason, a sizeable proportion of the effort of the research laboratory may be very profitably devoted to problems of control.

If a company maintains a research laboratory separate from the control laboratory, communications between the two and coordination of their efforts are often the most critical problems. To be effective, the people in the research group must be familiar with production, and have an appreciation of its problems. There should be no hindrance to the flow of ideas and suggestions between the men at the plant and those in the research laboratory. With these objectives in mind the following specific suggestions are offered for use in research planning:

1. Plan to include people on the research staff who are familiar with production as a result of first-hand experience with the manufacture or use of the Company's products.

2. Encourage the establishment of a substantial number of research projects concerned with problems of manufacturing control.

3. Maintain a close liaison with Technical Service and Quality Control units when these are not part of the research division.

4. Encourage communication at all levels between production and research personnel.

Like so much good advice, these suggestions are more easily made than effectuated, but you will agree no doubt that taken together they represent a very worthwhile program.

Acknowledgment

The present paper is based upon an analysis of numerous examples of the role of research in control suggested by the staff of The Fleischmann Laboratories and the Research Service Department of Standard Brands Incorporated. Their cooperation is gratefully acknowledged. The authors also wish to acknowledge the helpful advice received from Mr. Ferdinand P. Diemer.

Influence of Objective Quality Factors on Subjective Evaluation of Canned Sweet Corn[a,b]

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(Manuscript received August 28, 1952)

The smallest increments of several quality factors of canned sweet corn, measured objectively, which could be detected subjectively were determined. The minimum differences found were: 0.52% for moisture, 0.06% for pericarp, 0.95% for alcohol-insoluble solids and 0.68 ml. per 20 kernels for kernel volume. The relative influence of these quality factors upon the decisions of a panel of judges in determining the difference between lots of canned sweet corn was determined as 45% for canned moisture, 31% for pericarp, 22% for kernel volume, and 2% for alcohol-insoluble solids.

The actual components of quality of any foodstuff are either designated arbitrarily by judgement based upon experience and custom, or are proposed pursuant to laboratory studies. The limitations in the use of a single quality factor are readily recognized and generally admitted, but for simplicity, quality classification is frequently based upon a single factor. The integration of several factors is complex due to the additional time and expense involved in either objective evaluation, subjective evaluation, or in both. Assignment of the appropriate portion of the quality grade to each factor in determining a quality grade appears to be a difficult problem.

The subjective evaluation of food products for quality may be highly correlated with standard quality market grades. Kramer *(8)* reported on the relationship of objective tests, individually and collectively, with quality judging. Alcohol-insoluble solids content of canned corn samples alone and in comparison with ranking values had a correlation coefficient of —0.819. This value was slightly improved to 0.930 when pericarp and kernel size were considered in addition to the alcohol-insoluble solids. This high correlation provides no definite information on factors which influence the judges' decisions nor on the relative importance of these factors. Some measured factors actually may not influence the decisions of judges, but may be highly related to other factors which do have an influence. High correlation between pairs of objective quality factors may be of value in determining maturity and for maintaining quality of a food product, but of little value in understanding the effect of quality factors on subjective evaluation.

At present, objective tests of sweet corn maturity and quality are used to determine the chemical or physical changes occurring as maturity increases, or to determine specific characteristics of a given sample. The value of a test is determined by the correlation of the objective measurement with the results of a panel of competent judges in measuring the same quality or maturity characteristic. Kramer, Guyer and Ide *(9)* reported factors affecting the objective and organoleptic evaluation of quality in sweet corn. Moisture tests and succulometer tests were found to be very satisfactory in estimating the quality of raw sweet corn prior to cooking. After the corn was cooked, these tests were found to be good indicators of succulence and skin character, but not of flavor. The pericarp tests, determining quantity, were inferior to moisture and succulometer tests as an indicator of raw appearance or succulence, but equal to or perhaps slightly better for predicting skin character and flavor. This use of objective tests and the corresponding subjective evaluation does not take into consideration the limits of the smallest increment of quality change which can be detected by the average consumer.

The values of limits of an increment could be used to establish similar limits for corresponding objective tests, either chemical or physical, for quality. Mitchell and Lynch *(10)* in a subjective study of cream style sweet corn over a moisture range of 60 to 70% found by the use of triangular taste tests that a difference was not detected between 70.1 and 69.4%. Carter, Olson and Henry *(5)* referred to unpublished data which indicated that a difference in moisture content of 2% at harvest was generally the smallest that could be detected subjectively for Golden Cross Bantam corn.

Following the establishment of such limits for objective tests of quality of the canned product, corresponding limits of measurable quality increments could be placed on a practical objective test for the measurement of maturity changes of the raw product at harvest. A suitable test could then be devised which would be of sufficient accuracy and precision to indicate differences which subsequently would be detected subjectively. The multiple regression technique can be used for the determination of the relation of various quality factors of a product *(11)*. The relationship of various factors in determining an overall grade was computed by Calvin *(3)* for quality measurement of one variety of green beans. The computations were made on subjective evaluation data obtained by Kramer. Percentage weightings obtained by the use of the multiple regression technique were: 5 for color, 46 for maturity, 17 for fibrousness and 32 for flavor. For this variety the coefficient of determination was 0.90, which is exceedingly high. Recently Calvin *(4)* presented information on additional varieties. For the other varieties studied the weightings were different from those originally reported and the coefficients of variation ranged as low as 0.48. Kramer, Guyer and Ide *(9)* contended that the quality of sweet corn if fresh, frozen or canned was determined primarily by the degree of succulence, the quantity and toughness of the pericarp and flavor.

[a] Journal Paper No. J-2147 of the Iowa Agricultural Experiment Station, Ames, Iowa. Project No. 1052.

[b] Work on this project was made possible through a grant from the Iowa-Nebraska Canners Association.

[c] Present address: California Packing Corporation, Rochelle, Illinois.

The objectives of this investigation were: a, to determine the smallest increment of several variable quality factors of sweet corn, measured objectively, which can be detected subjectively by judges who would be considered more critical than the average consumer, and b, to determine the relative importance of the presently accepted objective quality measurements on subjective evaluation.

EXPERIMENTAL PROCEDURE

Twenty-eight sample lots of canned whole kernel sweet corn were prepared from plantings of the sweet corn variety Iochief during the season of 1952. Objective measurements of field and canned moisture content, pericarp content, alcohol-insoluble solids, and kernel volume were made to determine the quality composition of each lot. All computations were made on the basis of 4 replicates.

Standard procedures were employed for the measurement of moisture content *(1)* and alcohol-insoluble solids content *(6)*. The procedure used for the measurement of pericarp content was a modification of the method developed by Kramer, Guyer and Ide *(9)* and further modified by Gould, Krantz and Mavis *(7)*. The modifications in this procedure consisted of: a, use of a 50 g. initial sample blended with 100 ml. of water instead of a 100 g. sample blended with 200 ml. of water, b, use of the entire blended sample instead of a 0.20 aliquot, c, use of a 40-mesh screen instead of a 30-mesh screen, d, use of tared screens as modified by Gould, Krantz and Mavis *(7)* instead of obtaining tare weights after removal of the pericarp sample by flaming, e, employment of a 3-hour drying period instead of a 2-hour period. These modifications were used to obtain a representative value for pericarp content. A 50 g. sample could be utilized entirely instead of a 0.20 aliquot, as in the original procedure. The comminuted material formed a very poor suspension and aliquots did not appear to be uniform. The use of a tared screen was more feasible than flaming the dried pericarp and then weighing the screen. The use of a 3-hour drying period instead of a 2-hour period was necessary since a larger quantity of pericarp was retained on the filter screen due to the increase in the sample size. The volume of cut corn kernels was measured by water volume displacement. The sample consisted of 20 kernels obtained from a thoroughly mixed and drained can of corn. Adjoining kernels were used to avoid possible unintentional bias. The mixing of the corn kernels prior to selection provided a random sample. The corn kernels were placed in a 10-ml. graduated cylinder containing 5 ml. of water. The increase in volume caused by the addition of the 20 kernels of corn was the unit of measurement.

For subjective evaluation the triangle taste test *(2)* was considered the most desirable for use in detection of a difference between 2 samples.

Forty-two triangle tests were the minimum number submitted to each of the original judges during the period of acquainting the judges with the procedure and the product. The effects of visual comparisons were minimized by instructing the judges to avoid making such comparisons and by the employment of red lights in the judging booths.

RESULTS AND DISCUSSION

Relationships among the various objective measurements of all the sample lots studied were illustrated by the correlation coefficient values, Table 1. Perfect relationship would have a value of one and no relationship a value of zero. These values show that changes in these factors are closely related as sweet corn passes through the edible immature stage. This condition of high correlation among various objective measurements explains why several quality tests can be used interchangeably for judging maturity of sweet corn.

The initial approach to the problem of determining the smallest increments of objective quality which could be detected subjectively was to submit to the judges pairs of sweet corn samples which varied in moisture content. The judges were instructed to make their decisions on the basis of succulence only, disregarding other factors. Although an honest attempt appeared to be made to provide impartial judgements, comments were received that other factors such as toughness, starchiness, or flavor interfered while judging for succulence. A difference of 3.4% in moisture content was not detected on the second tasting period while, on the first and tenth periods, differences of 1.9 and 1.0% were detectable. Although these discrepancies could be the result of experimental variation, it was concluded that this was an unsound approach when the comments of the judges were taken into consideration.

TABLE 1

Relationship between pairs of objective quality measurements on sweet corn

Comparisons	Correlation Coefficients
Field moisture vs. canned moisture	0.884**
Field moisture vs. pericarp	0.904**
Field moisture vs. alcohol-insoluble solids	0.956**
Field moisture vs. kernel volume	0.637**
Canned moisture vs. pericarp	0.961**
Canned moisture vs. alcohol-insoluble solids	0.996**
Canned moisture vs. kernel volume	0.659**
Pericarp vs. alcohol-insoluble solids	0.959**
Pericarp vs. kernel volume	0.731**
Alcohol-insoluble solids vs. kernel volume	0.677**

** Significant at P = 0.01.

Judging on the basis of individual quality characteristics was considered impractical because of the unavoidable influence of other factors. As a result the judging procedure was modified, and subsequent judgements were made on the basis of any taste factor which would assist in discerning a difference.

The 3 objective quality factors—moisture content, pericarp content, and alcohol-insoluble solids content—were progressively increased in samples submitted to the panel of judges. A difference in the lots was not detected at the 95% significance level until the difference in moisture content was as great as 3.5%, in pericarp content 0.34%, and in alcohol-insoluble solids content 4.8% in the same comparison between 2 lots. When the alcohol-insoluble solids content was lowered to 4.1% while the moisture and pericarp contents were held constant at these levels, the difference between the 2 lots was still detectable. In another comparison of 2 lots the alcohol-insoluble solids content was 4.1% and the pericarp content was 0.36%. These values were greater than those in the 2 sets of samples formerly discussed; however, under these conditions the difference was not definitely detected with the moisture content at 2.9%. Although this result could be an indication that the degree of variation in moisture content required for detection of a difference lies between 2.9 and 3.5%, this point could not be proved adequately with the sweet corn lots available. The correlation among the objective quality factors of the samples was so high that sufficient variation did not exist to allow for determination of the influence of one factor independently of another. Subjective detection of differences among corn lots apparently was not based on a single quality factor, independent of all others, since a variation of 1.0% in moisture content indicated a detectable difference in the first experiment and a variation of 2.9 and 3.5% indicated a difference in the second experiment.

The multiple regression technique was used to determine the influence of objective quality factors on the decisions of the judges in discerning the taste difference between samples of corn. By this method the taste panel determined the difference between the 2 samples in a comparison irrespective of objective quality measurements. The number of correct solutions in 15 trials for each of 56 trials is listed in Table 2. In all cases in which 100% correct judgements were obtained the comparisons were not used, nor were they included in the 56 tabulated trials. Ten comparisons were made in which 100% correct judgements were obtained. If these data were used, the relationship of the dependent variable, the number of correct solutions, the independent variables, and the objective quality factors would not be informative; the independent variables could change without a corresponding change in the dependent variable.

TABLE 2
Objectively determined quality of canned sweet corn lots lots compared by subjective evaluation

Lot number	Objective measurement difference increments				Correct judgements per 15 tests
	Canned moisture	Pericarp	Alcohol-insoluble solids	Volume of 20 kernels	
	%	%	%	ml.	
1	1.0	0.06	1.4	0.2	6
2	1.9	0.16	2.9	0.5	9
3	1.6	0.18	1.9	0.9	10
4	2.4	0.23	2.7	0.9	11
5	0.9	0.08	0.8	0.7	12
6	0.5	0.07	0.2	0.4	12
7	1.5	0.13	1.2	0.6	13
8	0.1	0.05	0.0	0.8	4
9	0.6	0.05	1.1	0.2	7
10	0.3	0.06	0.1	0.0	7
11	1.1	0.02	1.6	1.6	9
12	0.5	0.07	0.2	0.7	11
13	0.7	0.00	2.0	0.7	11
14	0.4	0.01	1.0	0.0	10
15	0.3	0.06	0.6	0.5	11
16	0.7	0.10	1.1	0.2	7
17	1.4	0.04	1.5	1.8	9
18	1.5	0.06	1.7	0.2	10
19	1.0	0.12	1.4	0.4	13
20	2.2	0.16	2.8	0.0	8
21	2.5	0.18	3.1	0.2	13
22	3.2	0.28	4.2	0.4	14
23	4.6	0.32	5.7	2.0	13
24	4.8	0.34	4.9	1.3	14
25	0.5	0.06	1.0	0.3	11
26	1.0	0.04	1.7	1.1	11
27	0.0	0.08	0.3	1.5	10
28	1.3	0.02	2.0	0.4	6
29	0.2	0.04	0.3	0.2	7
30	0.1	0.07	0.1	0.3	8
31	0.8	0.04	1.2	0.1	9
32	0.9	0.02	1.6	1.6	11
33	0.1	0.02	1.1	0.2	7
34	0.9	0.10	0.3	0.2	9
35	0.1	0.06	1.4	1.3	8
36	0.3	0.06	0.6	0.5	9
37	1.2	0.07	1.8	0.0	11
38	0.4	0.01	1.0	0.0	10
39	1.3	0.05	0.7	0.2	11
40	0.2	0.03	1.3	0.2	12
41	0.7	0.03	0.8	0.2	9
42	1.0	0.03	0.3	0.4	13
43	0.4	0.01	0.3	0.7	8
44	1.8	0.07	1.8	0.1	9
45	1.4	0.06	1.5	0.0	5
46	0.4	0.08	0.5	0.7	9
47	0.6	0.04	0.9	0.3	8
48	0.4	0.00	0.8	0.8	7
49	0.9	0.10	1.5	0.3	6
50	0.3	0.06	0.6	0.0	5
51	0.9	0.04	0.9	0.8	3
52	0.9	0.08	1.4	0.1	8
53	0.3	0.02	0.2	0.2	6
54	0.4	0.08	1.3	0.0	8
55	0.5	0.06	0.5	0.4	5
56	0.6	0.02	0.3	0.8	8

The multiple regression equation computed from the 56 comparisons presented in Table 2 is:

$$\hat{Y} = 7.64 + .647\ X_{cm} + 6.038\ X_p + .027\ X_a + .622\ X_v. \quad (1)$$

The symbol designations are:

$\hat{Y}$ = estimated number of correct solutions.

X_{cm} = difference in percent moisture between two lots being tested.

X_p = difference in percent pericarp between two lots being tested.

X_a = difference in percent alcohol-insoluble solids between two lots being tested.

X_v = difference in volume of 20 kernels, expressed in milliliters, between two lots being tested.

The coefficient of multiple regression, R, which measures the degree of linear association between the estimated and observed values of correct solutions was 0.476. This value was significant at the 99% probability level. The square of the coefficient gives the proportion of the sum of squares of the dependent variables accounted for by the multiple regression. This value, the coefficient of determination, R^2, was 0.227. Actually this linear multiple regression explained only approximately 23% of the variability in the decisions of the members of the judging panel.

Additional proof that a significant proportion of the variation involved in the detection of differences in sweet corn samples was accounted for by multiple regression was the significant F value obtained by analysis of variance, Table 3. This F value was significant at a probability level beyond 99%.

TABLE 3
Analysis of variance for the variations of the multiple linear regression of the subjective evaluation data

Source of variation	Degrees of freedom	Sum of squares	Mean squares
Deviations due to regression	4	85.009	21.252**
Deviations about regression	51	289.110	5.669

** Significant at P = 0.01.

A possible explanation for the low coefficient of determination value is that the 4 quality measurements studied collectively account for this portion of the variation, and in order to account for the additional variation, other factors are necessary. Additional factors which could influence the decisions of the judges would not individually be expected to account for a greater share of the variation in the dependent variable than any one of the objective characteristics studied. For this assumption the number of additional factors would be large.

Since quality, Q, may be considered as the sum of characteristics of a given food product, it may be represented in mathematical symbols in the form of an equation like the regression equation developed, where

$$Q = \beta_0 + \sum_{i=1}^{n} \beta_i X_i. \quad (2)$$

with β_o equal to Q when X_i equals zero and β_i equal to the change which occurs in Q for a unit change in X_i. With the factors studied,

$$Q = \beta_o + \sum_{i=1}^{4} \beta_i X_i \qquad (3)$$

With additional factors,

$$Q = \beta_o + \sum_{i=1}^{4} \beta_i X_i + \sum_{i=5}^{n} \beta_i X_i \qquad (4)$$

The factors contained in the last term of equation (4) represent the characteristics not included in this study. The factor, X_i, may be a pure number or function of pure numbers, as an exponential or product of 2 or more pure numbers.

The standard partial regression coefficients, Table 4, express the relative importance of the various independent variables in accounting for the variation in the dependent variable. Thus, canned moisture content accounts for the largest proportion of the variation in the dependent variable, or approximately 45% of the total. Similarly, pericarp accounts for approximately 31%, kernel volume approximately 22% and alcohol-insoluble solids only approximately 2% of the variation.

TABLE 4
Relation of objective measurements to subjective evaluation on an independent basis

Objective measurement	Partial regression coefficients	Standard partial regression coefficients	Standard partial regression coefficients expressed as percent of total
Moisture	0.647	0.243	44.58
Pericarp	6.038	0.171	31.38
Alcohol-insoluble solids	0.027	0.012	2.20
Kernel volume	0.622	0.119	21.83

It may be concluded from this that moisture is the best criterion for judging quality in sweet corn among the objective measurements of pericarp, kernel volume, and alcohol-insoluble solids.

It is possible to determine the amount of each quality factor, measured objectively, required to influence the judges such that a difference between 2 samples can be detected. Regression equation (1),

$$\hat{Y} = 7.64 + .647\ X_{cm} + 6.038\ X_p + .027\ X_a + .622\ X_v,$$

can be used for these determinations.

Although the regression coefficients were not statistically significant at the 95% probability level, it appeared logical that various quality factors, as measured objectively, must influence subjective decisions. With this assumption it was felt that these regression coefficients could justifiably be used for determining the differences in objective measurements required for subjective difference detection.

For distinguishing between 2 lots of corn 9 correct judgements per 15 tests were required at the 95% probability level to ensure that a detectable difference existed between the 2 lots under investigation. Assuming this probability level, several statements can be made relative to the above equation: a, the dependent variable, $\hat{Y}$, must equal at least 9 if 15 tests are conducted; b, the first term on the right side of the equation should equal 5 if all quality factors had been studied, since 5 correct solutions would occur by chance; c, the difference between the first term, a constant, and the dependent variable, $\hat{Y}$, having a value of 9 in this example, equals the quantity required to be accounted for by the independent variables, X_i's, in equation (1), and d, a change in any X_i, with all other X_i's held constant, necessary to make the dependent variable, $\hat{Y}$, equal to 9 is the difference increment in the objective quality measurements which can be detected subjectively by the judges who were on the panel used to obtain the regression equation empirically.

The computed differences at the 95% level, necessary to ensure detection between canned sweet corn lots for each variable measured objectively and with all others held constant are listed in the first column of figures in Table 5. The values obtained were: 2.10% for canned moisture, 0.23% for pericarp, 50.37% for alcohol-insoluble solids, and 2.19 ml. per 20 kernels for kernel volume.

When the extreme values of the 95% confidence limits were used for computation of differences in objective measurement, a range as listed in the second column of Table 5 exists. The lower confidence limit is negative which means that the population regression coefficient could have a value of zero. Should this be the case, the increments which would allow detection would be infinite in size. However, the objective of this investigation was to ascertain the smallest increment between objective measurements which can be detected by a panel of competent judges. These minimum differences are the smaller values in the second column. These values—0.52% for moisture; 0.06% pericarp; 0.95% for alcohol-insoluble solids; and 0.68 ml. per 20 kernels for kernel volumes—are the minimum values which would be detected.

TABLE 5
Objective measurement difference increments required for detecting quality differences subjectively

Objective quality factors	Increments	
	Required difference	Confidence limits at 95% probability level
Canned moisture content	2.10%	0.52 to ∞ %
Pericarp content	0.23%	0.06 to ∞ %
Alcohol insoluble solids content	50.37%	0.95 to ∞ %
Volume per 20 kernels	2.19 ml.	0.68 to ∞ %

In order to determine the feasibility for prediction of results in the subjective detection of differences in sweet corn lots, 19 triangle tests were conducted. Multiple regression equation (1),

$$\hat{Y} = 7.64 + .647\ X_{cm} + 6.038\ X_p + .027\ X_a + .622\ X_v,$$

was used for the prediction of the number of correct judgements in 15 triangle tests for 19 lot comparisons. The lot comparisons used were combinations of corn lots not used in the computation of the multiple regression equation (1). The actual number of correct judgements and the predicted number of correct judgements with 95% confidence intervals were tabulated for comparison in Table 6.

TABLE 6
Predicted and actual number of correct judgments in distinguishing lots of canned sweet corn

Lot number	Objective measurement difference increments				Correct judgements per 15 tests		
	Canned moisture	Pericarp	Alcohol-insoluble solids	Volume of 20 kernels	95% C.L.	Actual	Deviation
	%	%	%	ml.			
1	0.6	0.06	0.6	0.6	8.78±4.90	8	0.78
2	1.0	0.19	1.3	0.3	9.66±5.21	6	3.66
3	1.6	0.16	2.6	0.9	10.27±4.59	12	−1.73
4	0.1	0.00	0.5	0.1	7.78±4.91	9	−1.22
5	2.6	0.29	2.7	0.6	11.52±5.32	10	1.52
6	2.4	0.18	2.8	1.9	11.53±6.11	14	−2.47
7	1.9	0.10	2.6	0.9	10.10±5.40	11	−0.90
8	3.4	0.17	3.5	0.3	11.15±5.41	10	1.15
9	4.4	0.26	6.2	0.5	12.54±5.72	12	0.54
10	4.4	0.29	5.5	0.0	12.39±5.59	13	−0.61
11	3.5	0.12	5.4	0.1	10.84±5.82	10	0.84
12	4.0	0.18	4.9	0.5	11.76±5.55	12	−0.24
13	3.8	0.32	4.3	1.0	12.77±5.35	13	−0.23
14	3.6	0.22	5.0	0.6	11.81±5.17	12	−0.19
15	1.0	0.13	0.8	0.7	9.53±4.97	11	−1.47
16	4.6	0.32	5.8	0.7	13.14±5.52	13	0.14
17	3.6	0.35	4.5	0.8	12.70±5.45	14	−1.30
18	3.1	0.29	3.5	0.8	11.99±5.19	11	0.99
19	0.0	0.06	0.9	0.6	8.40±5.06	8	0.40
Mean					10.98	11.00	

Correlation coefficient (r): Predicted vs. Actual: 0.760**

** Significant at P = 0.01.

The relationship between the actual and predicted results was expressed by the correlation coefficient, $r = 0.760$, which was statistically significant beyond the 99% probability level. For all 19 observations the means for the predicted and actual correct decisions were 10.98 and 11.00 respectively in 15 triangle tests. There was no statistical or practical difference between the means of the two methods for the determination of a difference between samples. The individual lot comparisons are of more interest from a prediction point of view. At the 95% probability level, the confidence limits allow for a deviation of approximately 5 correct judgements; however, actually the greatest deviation obtained was 3.66, well within the confidence limits.

The decisions of the judges were highly variable for any one set of triangle tests. The individual results for the 56 sets of triangle determinations used to determine the regression equation (1) were analyzed to determine if the judges in this experiment varied in their ability. The means for each judge over all 56 lot comparisons varied from 1.62 to 2.16. However, by use of the analysis of variance technique the difference in ability to make correct judgements was not verified at the 95% probability level, Table 7. This inability to detect a difference among judges may be due to the variation in the results for each lot comparison and may also account for the absence of statistically significant partial regression coefficients.

Since each judge participated in 3 triangle tests at each sitting, there was the possibility of fatigue. The mean number of correct judgements for the first, second and third trial at a sitting were 3.05, 3.07 and 3.00, respectively. The differences among the number of correct judgements rendered at the first, second and third trial at a sitting were not significant at the 95% probability level, Table 8. This indicates, from a practical point of view, that 3 triangle tests per judge were not too fatiguing at one sitting.

TABLE 7
Analysis of variance for the variation among the decisions of the judges

Source of variation	Degrees of freedom	Sum of squares	Mean squares
Lots compared	55	72.9	1.32
Judges	4	6.1	1.52
Exp. error	220	150.5	0.68

TABLE 8
Analysis of variance for the variation among orders of taste trials

Source of variation	Degrees of freedom	Sum of squares	Mean squares
Lots compared	55	124.709	2.267
Order of trials	2	0.155	0.078
Exp. error	110	141.845	1.288

SUMMARY AND CONCLUSIONS

The influence of quality factors other than those under study affected the decisions of the judges to such an extent that differences between lots could not be distinguished by a single quality factor.

Objective quality factors were so highly correlated that one factor could not be varied independently of other factors sufficiently to obtain all desired combinations for a study of the influence of objective quality factors on subjective evaluation.

An equation representing the effects of the 4 objective quality measurements on the subjective decisions of the judges was formulated by the use of the multiple regression technique. Paired sweet corn lots were submitted to the panel of judges for detection of a difference on any desired palatability basis.

The degree of linear association among the dependent and the independent variables was statistically significant at the 99% probability level since the coefficient of multiple regression, R, and the F value in an appropriate analysis of variance were significant at that level. The portion of the sum of squares of the dependent variable accounted for by the independent variables was approximately 23%. While 4 factors accounted for 23% of the variation in number of correct judgements rendered

by the panel, they were the commonly accepted quality measurements and it was considered very likely that a large number of additional factors were involved in influencing the subjective decisions of the judges.

Relative effects of the objective measurements were determined by computation of the standard partial regression coefficients between the objective measurements and the correct judgements of the panel. The proportionate effect was: canned moisture 45%, pericarp 31%, kernel volume 22%, and alcohol-insoluble solids 2%. It was concluded that moisture was the best criterion for the judging of quality in sweet corn among the objective measurements of pericarp, kernel volume, and alcohol-insoluble solids.

The minimum difference in objective quality measurements which could be detected by the experienced panel of judges in this investigation at a 95% probability level was: 0.52% for moisture, 0.06% for pericarp, 0.95% for alcohol-insoluble solids and 0.68 ml. per 20 kernels for kernel volumes. It may be concluded that these minimal difference increments could be the basis for determining the accuracy required for measurement of objective quality factors.

Verification of the regression equation for prediction of establishing a difference between sweet corn lots was checked on 19 separate triangle taste tests. All the actual values of correct judgements fell within the 95% confidence limits of the predicted results.

The ability for detecting differences between lots of sweet corn did not vary among the judges. It was also found that 3 triangle tests at one sitting was not fatiguing for the judges.

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The Removal of Excess Copper and Iron from Wine[a]

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The removal of excess copper by precipitation with rubeanic acid and the removal of excess copper and iron by treatment with a Prussian blue - ferrocyanide adsorption complex is described. Data on other possible substitutes for soluble ferrocyanides are given as well as conditions limiting their application.

The formation of undesirable turbidity and sediments in California wines containing excessive amounts of copper and iron is one of the major problems of the industry. Presence of these metals as ions or complexes has resulted in not only undesirable changes in appearance but also in the undesirable changes in flavor. This condition has existed in the California wine industry since repeal and is common to all wine-producing regions. The tolerance of wine for metals and the conditions under which the white ferric phosphate casse, the blue ferric tannate casse and the white to reddish-brown copper casse will form have been described in the publications of Amerine and Joslyn *(1, 2)*, Joslyn and Amerine *(27)*, Castella *(11)*, Cruess *(15)*, Marsh *(30)*, Waal *(55)*, and others. The most extensive investigations of the factors influencing the distribution of iron and copper ions in wine in various states of oxidation and combination and of the conditions affecting formation of their colloidal suspensions and deposits were those of Ribéreau-Gayon *(43-47)*.

Ribéreau-Gayon *(43, 44)* and others have shown that under reducing conditions, the iron in wine exists in the ferrous state largely as free Fe^{++} and to a smaller extent in soluble organometallic complexes. On contact with air, the ferrous ions are rapidly oxidized to the ferric state in which they can form colloidal suspensions or precipitates of ferric tannates (blue or ferric casse), or of the very slightly soluble ferric phosphate (white casse). Phosphate casse, however, occurs only in the region of pH 2.9-3.6. The ferric ions however readily form complexes with citric acid and other organic acids.

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[b] Senior Laboratory Technician on the Wine Advisory Board research grant on metals in wine from February, 1949 to June 30, 1952.

[c] Senior Laboratory Technician on the Wine Advisory Board research grant on metals in wine from July 1, 1948 to December 30, 1949.

Some data on the distribution of iron in California wines, using the procedures established by Ribéreau-Gayon *(43, 44)*, have been reported by Marsh and Nobusada *(31)*. Large changes in total iron content are known to occur in wines exposed to air during fermentation, storage, and refrigeration. The copper in wine under reducing conditions forms colloidal cupric sulfide. Ribéreau-Gayon *(43, 46)* postulated a preliminary reduction of Cu^{++} by some reducing agent to Cu^{+}, then reduction of sulfite to H_2S by Cu^{+} and formation and subsequent flocculation of colloidal cupric sulfide. Copper is apparently necessary for the reduction of sulfite in wine. Whether the white cloudiness which forms in wine in the presence of excess Cu^{++} under reducing conditions is due only to formation of CuS is not known nor is it known that the deposit, which readily dissolves on aeration or oxidation is only CuS. Turbidity in wines susceptible to copper casse stored in absence of air is prevented by heating or by addition of a stabilizing colloid like gum arabic. Flocculation of the finely dispersed colloidal cupric sulfide occurs as a result of the action of electrolytes present and the wine becomes turbid and finally a deposit of reddish-brown cupric sulfide containing occluded organic matter (largely proteinaceous) forms.

It is now known that the state of oxidation or reduction of the wine, its pH value, its content of sulfur dioxide, of organic acids capable of complexing ferric ions (citric, malic, tartaric), or tannins, anthocyanin and related flavonols, of phosphate and sugars and sugar derivatives, and other constituents, all influence the tolerance of the wine for metals. Failure to control these variables led Searle, *et al. (52)* to report that the tolerance of wines for metals is between 1-3 p.p.m. for iron and tin, 15-30 for chromium, 60-100 for copper, 100-200 for aluminum, and over 200 p.p.m. for nickel and zinc. Mrak, *et al. (36)* reported the tolerance of California chablis and sauterne white table wines to be 2 p.p.m. for aluminum, chromium, copper and iron, 5 p.p.m. for tin and zinc, and 50 p.p.m. for nickel. Jaulmes *(24)* recently reported that French wines will not be susceptible to iron casse below 12 to 15 mg. of iron per liter for white wines and 20 to 25 mg. per liter for red wines. Copper casse in French wines will occur at 0.5 mg. of copper per liter.

In 1940, the tolerance of sulfited white table wines for copper was suggested by Amerine and Joslyn *(1)* to be between 0.5-0.8 p.p.m. for table wines and about 0.5 p.p.m. for dessert wines *(27)*. Since then, copper casse has been aggravated a, by the bottling of wine at the winery and shipment of wine in the bottle rather than in bulk; b, by development of better closures for wine bottles which, while preventing losses in quality by leakage and oxidation, result in the production of reducing conditions favorable to the formation of copper casse; c, by storing wines in the bottle at the winery or the bottling plant; and d, by the increase in the period of time between bottling and consumption during which the wine is expected to remain bottle bright. At the present time the general consensus of the industry supports the conclusions of Nasledov *(37)* that for a California wine to be metal stable it must contain less than 0.2-0.3 p.p.m. of copper and 3-4 p.p.m. of iron.

The widespread use of equipment and fittings of brass, bronze or other copper alloys in the industry, originally done to minimize contamination of wine with iron, and the reduced tolerance of wines to copper has focussed attention on copper pick-up. Iron casse has been controlled by lessened exposure to iron-bearing metals and by the use of citric acid as stabilizing agent so that at present the chief cause of metal clouding is excess of copper. Data on copper content of California grapes and on the extent of copper pick-up during crushing, fermenting, pumping, filtering and bottling were not available until recently. The development of a rapid sensitive method of determining copper by Marsh *(29)* finally led to its application to determination of changes in copper content of musts and wines by Nasledov *(37)*. The latter reported that new wines less than 5 months old are practically copper free, but the nearly finished wines may contain from 1.4 to 2.0 p.p.m. of copper. He also showed that this excess of copper was picked up by contact of the wine with pasteurizers, pumps, chilling equipment, filters or filling machines constructed in part or entirely of brass or other copper alloys. The increase in copper content of wines was reported to be 0.5 p.p.m. for pumping, 1.7 p.p.m. for filtering and 0.6 p.p.m. for bottling.

At present the most efficient method known for reducing the iron and copper content of wine is by "blue-fining," i.e., precipitation by the addition of potassium ferrocyanide under carefully controlled conditions. When properly used, under conditions that eliminate any possibility of the presence in wine of uncombined ferrocyanide, blue-fining will effectively remove both copper and iron without adversely affecting the palatability of wine and will even stabilize the wine and improve it, Couerbe *(14)*. Although ferrocyanides have been used for many years in the removal of heavy metals from wines, Fessler *(20)*, Heide *(21)*, Miconi *(34)*, Ribéreau-Gayon *(45)*, Saywell *(50)* and Waal *(55)*, vinegars, Saywell *(49)*, organic acids and other products, Brown *(6)*, the conditions of their use were developed largely by empirical procedures without due consideration of the peculiar electronic field forces of the molecules. Our knowledge of the physical-chemistry of ferrocyanides has been greatly expanded in recent years, Cane *(10)*, Davidson *(16, 17)*, Weiser *(58, 59)* but this has not been applied as yet in its use.

Although blue-fining has been used extensively in Germany under strict government supervision, other countries have been reluctant to use it because of the possible hazards involved in the use of a cyanogenetic compound. There has been great interest in finding a substitute for ferrocyanide. A suitable substitute should be preferably a precipitant for heavy metals which would remove them efficiently, be capable of use by direct addition to the wine without the introduction of additional equipment or operations other than those normally used in fining and filtration, be perfectly safe to use and, finally, should be as economical. There have been many attempts to develop suitable substitutes. Aeration with or without refrigeration and fining to remove excessive amounts of iron has not been successful because it adversely affected the quality of wine and did not accomplish the removal of copper; precipitation with sodium sulfide, suggested by Ribéreau-Gayon *(46)*, while it reduced the copper content also impaired

the quality of wine; the addition of alpha cellulose, Ramage *(41)*, of lignic acid, Nelson, *et al. (39)*, and of calcium phytate, Cohee and Steffen *(13)*, as iron binding or precipitating agents has been suggested; natural or synthetic ion exchange resins have been proposed, Felton *(18)*, Ramage *(40)*, Trevor *(53)*. In recent years as a result of a better understanding of the chemistry of organo-metallic compounds, particularly the metal chelate compounds, Johnson *(25)*, Martell and Calvin *(32)*, attention has been paid to the possible uses of chelating compounds for binding or separation of metals. Such compounds have already been introduced for deactivating copper in the catalytic deterioration of gasoline during storage *(38, 56)*, and in water softening and other uses *(3)*. At the time our investigations were begun, Versene, ethylene diamine tetra acetic acid tetra sodium salt, was already in use as a powerful sequestering agent for metallic ions *(3, 5)*, and was suggested for use in wine by its manufacturers. Krum and Fellers *(28)* reported on clarification of wine by this sequestering agent. We had proposed to use chelating compounds for metal removal either as such or in the form of specific tailor-made cation resins prepared by adsorbing them upon the surface of an inert carrier. Such ion exchange resins are now being manufactured and tested, Calvin *(8)*. The results of our investigations on metal removal from wine are presented briefly here and will be discussed in more detail elsewhere.

EXPERIMENTAL PROCEDURE AND RESULTS

Survey of possible organic chelating agents as metal precipitants. In the preliminary survey of possible substitutes for ferrocyanides which might form insoluble metallic derivatives, a white table wine made of French Colombard grapes in Martinez was used. This wine of the vintage of 1947 was racked in January 1948 and gelatin fined and filtered in August 1948 when it was obtained for our investigations. It was stored in full 5 gallon carboys at 50° F. (10° C.) until used, care being taken to avoid aeration. Its analysis by the methods given in Amerine and Joslyn *(1)* was as follows:

Alcohol, vol. %	12.28%
Total acidity as tartaric	0.66%
Volatile acidity as acetic	0.045%
Reducing sugar as glucose	0.14%
Total sulfur dioxide	167 p.p.m.
Tannin	0.050%
pH (at 21° C.)	3.55

Copper determinations were made by the sodium diethyldithiocarbamate colorimetric method, using the modification of Marsh *(29)*. The standard copper solution was made by dissolving pure electrolytic copper in nitric acid according to the procedure outlined by Sandell *(48)*. Readings were taken on a Klett-Summerson photoelectric colorimeter using a green filter (540 milli-microns).

The total iron determinations were carried out by the colorimetric method using ortho-phenanthroline, Saywell and Cunningham *(51)*, Hummel and Willard *(28)*. The standard iron solutions were prepared by dissolving 99.8% pure iron wire in dilute nitric acid. All readings were made using the Klett-Summerson colorimeter with a purple filter (420 milli-microns).

The distribution of the iron content of the wine into ferric, ferrous, and complex iron was made by the procedure of Marsh and Nobusada *(31)*. Ferric ion was determined by means of a standard potassium ferrocyanide solution. Then total ferric plus ferrous ion was determined similarly after the addition of hydrogen peroxide to oxidize the ferrous to ferric ion. Hence, with the total iron already determined, the concentration of each constituent was calculated as follows:

$$[Fe^{+++}] + [Fe^{++}] + [\text{Fe complex}] = 5.8 \text{ p.p.m.}$$
$$[Fe^{+++}] = 0.7 \text{ p.p.m.}$$
$$[Fe^{+++}] + [Fe^{++}] = 2.3 \text{ p.p.m.}$$

Therefore: $[Fe^{+++}] = 0.7$ p.p.m., $[Fe^{++}] = 1.6$ p.p.m., and [Fe complex] = 3.5 p.p.m.

Although the original copper content of the wine was only 0.7 p.p.m., additional copper was added to provide samples containing 1, 2, 4, and 6 p.p.m. copper, all of which contained 5.8 p.p.m. iron.

Wine samples ranging from 25 to 40 ml. were treated with a number of possible chelating reagents added either stoichiometrically or in definite excess. After approximately 48 hours, "Super-cel" was added with agitation and each sample filtered through No. 2 Whatman paper in glass funnels. Appropriate samples for the iron and copper determinations were then withdrawn from the filtrates and the analyses carried out according to the procedures mentioned above.

Excesses of reagent were also checked for in the cases of rubeanic acid, the Prussion blue-potassium ferrocyanide, and ordinary potassium ferrocyanide treatment. This was accomplished by merely adding copper ion in the case of rubeanic acid and ferric ion in the latter 2 treatments involving possible ferrocyanide ion excess.

As shown in Table 1, of all the organic compounds tried only rubeanic acid (dithio oxamide) precipitated excess of copper. This compound effectively, and practically quantitatively, precipitates copper ion when used in stoichiometric proportion (both copper and iron considered in calculation of theoretical quantity of reagent required). The precipitation seems to proceed slowly enough to assure relatively large particle size (in contrast to the blue-fining process) and filtration or decantation of the black precipitate was accomplished readily.

The results indicate that the iron content is only slightly reduced by rubeanic acid. It is possible that rubeanic acid precipitates only the iron not bound in complex form or that the solubility of iron rubeanate is too high to reduce the concentration below the indicated minimum value (4-6 p.p.m.).

Besides potassium ferrocyanide, the only other satisfactory results were obtained with the use of the Prussian blue powder containing an excess of ferrocyanide ion. This substance, based on the extremely high adsorptive power of Prussian blue, reduced the copper content to practically zero in all cases and, in addition, shows possibilities of eliminating iron also. In one case the iron content was reduced to practically zero, but in other cases, in which decreased amounts of the powder were used, the iron was only partially removed or not at all.

Another difficulty encountered in the first runs was that a slight trace of ferrocyanide ion was imparted to the wine; however, a sample of the Prussian blue powder, after rewashing thoroughly with warm water, effected removal of copper and partial removal of iron without indication of ferrocyanide being introduced.

The fact that this powder contains iron in its "inert-carrier" (Prussian blue) and also in its active component (ferrocyanide ion) and yet does not increase the iron concentration—actually lowering it in some cases—is indicative of a sound theoretical basis for this type of reagent. Theoretically this amounts to an ion exchanger whereby traces of the heavy metal can be extracted from their solutions and replaced with potassium (or possibly hydrogen) without the introduction of any other soluble constituent.

Another possibility along this line is the possible use of a coagulated protein such as edestin or albumin as a carrier for ferrocyanide ion. It is known, Hendrix *(22)*, that these amphoteric substances are capable of com-

TABLE 1

Precipitation of iron and copper in white table wine by various chelating agents. (Initially, wine contained 2 p.p.m. of copper and 5.8 p.p.m. of iron)

Reagent	Equivalents of metal ion chelated per mol.	Solution added	Residual metal content, p.p.m.	
			Copper	Iron
Alpha benzoinoxime	1	0.10mM per ml. 95% alcohol	2.0	9.2*
Dithizone	1	0.039mM per ml. 95% alcohol made alkaline with NH_3	*	7.1*
Schiff base of salicylaldehyde	2	0.05mM per ml. 95% alcohol	2.1	5.3
Schiff base of sodium salicylaldehyde 5 sulfonate	2	0.04mM per ml. 95% alcohol	1.7	6.4
Sodium ethylene diamine tetra acetate (analytical grade Versene)	1 (Atom)	0.02 mM per ml. water made alkaline with NH_3	1.7	6.4
Thenoyl trifluoro acetone[1]	1	0.1mM per ml. 65% ethanol	1.8	6.4
Phthalocyanine	1 (Atom)	Added as solid and in concentrated H_2SO_4	2.0	7.2
Rubeanic acid[2]	1 (Atom)	0.01mM per ml. absolute alcohol	0.0-0.1	4.7-5.5
8 Hydroxyquinoline	1	0.01mM per ml. 95% alcohol	1.4	5.0
Quinaldinic acid	1	0.01mM per ml. 95% alcohol	1.4	5.8-8.4
Salicylaldoxime	1	0.01mM per ml. 95% alcohol	1.4	5.6-7.7
Alpha nitroso beta naphthol	1	0.01mM per ml. 95% alcohol		6.9
Cupferron	1	0.01mM per ml. 50% alcohol	1.5	2.9-5.3
Tannic acid		0.01mM per ml. 10% alcohol	2.0	7.8
Potassium ferrocyanide[3]		0.0025mM per ml. water	0.0-0.1	2.6-3.1
Prussian blue—ferrocyanide[4]		Added as solid	0.0	0.5-9
Diatomaceous earth[5]		Added as solid	1.5-1.8	5.4-8.3
Bauxite, activated[6]		Added as solid	2.0	6.7-7.2

[1] Chelating agent introduced by Calvin *(9)*.

[2] Selected because it was reported to form insoluble compounds with copper and other metals, Ray and Ray *(42)* and its copper complex was used in colorimetry, Center and MacIntosh *(12)*.

[3] $K_4Fe(CN)_6.3H_2O$, M. W. = 422.4 was used. It was assumed in calculating equivalent weight that only 3 of the 4 K's are replaceable.

[4] As a result of our survey of the chemistry of heavy metal ferrocyanides, it appeared likely that an adsorption complex of potassium ferrocyanide would be useful, particularly because of the ease with which ferrocyanide ion as well as cations are adsorbed by Prussian blue, Weieser *(58)*, McBain *(33)*. Ramage *(40)* proposed the use of a ferrous ferricyanide preparation precipitated in the presence of excess soluble ferricyanide, either alone or in combination with iron oxide gel, silicia gel, activated carbon or cellulose. Our preparation was made by adding a 50% excess of potassium ferrocyanide solution, over the stoichiometric requirement, to ferric chloride solution. After washing thoroughly with several batches of water and drying in the vacuum oven, the resulting powder was ground in a mortar and mixed with approximately 10% by weight of Super-cel. The latter step provided nuclei for the relatively minute Prussian blue particles to adhere to, thus increasing the effective particle size. This was conducive to more rapid settling and better filtration in the subsequent wine treatment. The formula for the above powder was assumed for the purpose of calculation to be $KFe'''Fe''(CN)_6 \cdot 1/3K_4Fe(CN)_6$ with a molecular weight of 430. This reagent was added in proportions ranging from 0.2 to 1.0% corresponding to excesses of ferrocyanide ranging from 5 to 1000%. It was assumed that only 3 K's of $K_4FE(CN)_6.3H_2O$ are replaceable.

[5] Since diatomaceous earth, Hyflo Super-Cel grade made by Johns Manville was used with all the above reagents it was included as a blank control.

[6] 0.16-0.19 g. of bauxite per 40 ml. of wine were used.

* Interfering coloration or cloudiness not removable by filtration.

bining with anions when on the acid side of the isoelectric point. The following equations illustrate, in general, what could be expected to take place:

$$\text{R-CH-COO}^- + \text{H}^+ \longrightarrow \text{R-CH-COOH}$$
$$\quad\quad\text{NH}_2 \quad\quad\quad\quad\quad\quad\quad\quad\quad\quad\text{NH}_3^+$$

$$4\,\text{R-CH-COOH} + \text{Fe(CN)}_6^{4-} \longrightarrow$$
$$\quad\quad\text{NH}_3^+$$
$$\left[\begin{matrix}\text{R-CH-COOH}\\ \text{NH}_3^+\end{matrix}\right]_4 \text{Fe(CN)}_6^{4-}$$

In this way the ferrocyanide becomes available in soluble form only in the presence of heavy metal ions, and the danger of its contamination of wines is considerably lessened.

It is also noteworthy that in the copper determinations, conducted without preliminary ashing of the wine, that the high copper recovery obtained with a variety of chelating agents including Versene, indicates that these do not bind cupric ion as effectively as dithizone.

Calcium Phytate as a Heavy Metal Precipitant

Several naturally occurring substances are known to react with and thus possibly remove iron and copper salts. Lignic acid has been reported by Nelson *et al. (30)* to remove iron from water solutions; chlorophyll is known to exchange its Mg. for Cu. but so far has not been tried either as such or in the form of chlorophyllins and related derivatives; phytic acid as calcium phytate was suggested for the removal of heavy metals from vinegar *(13)*. Since phytic acid was not available, the applicability of calcium phytate to wine was tested, essentially under the conditions proposed for vinegar. Whether the pH was low enough or the periods of treatment long enough for the reaction to occur was not determined at this time.

Procedure. Equivalent amounts of solid calcium phytate were added to the white wine used previously, adjusted to various concentrations of copper at a constant iron concentration. Samples were allowed to stand at 45° C. for 120 hours. Twenty ml. samples were removed at various intervals, filtered and analyzed for copper and iron. The calcium phytate was added to 200 ml. portions of wine in the following amounts:

Cu	Fe	Calcium phytate
p.p.m.	p.p.m.	grams
0.6	8.1	0.007
5.0	8.1	0.010
10.0	8.1	0.015

The results obtained are shown in Table 2. Under our conditions, this reagent was found to be effective in only reducing the iron content but removed little or no copper.

Removal of Metals by Ion Exchange Resins

Several selected ion exchange resins were investigated to determine their exchange capacity for iron and copper. Duolite C-3, Dowex-50, Monsanto L 1189B, Zeo Rex and Permutite K cation exchange resins were used both in hydrogen and in the potassium cycle. In addition Duolite A-3 and Amberlite IR-4B anion exchange resions were used in the tartrate cycle. Typical of the data obtained for the cation exchange resins is that for Dowex-50 shown in Table 3 and Figures 1 and 2, for 30 ml. of resin at a flow rate of about 5 ml. per min. of French Colombard wine, adjusted to 2.2 p.p.m. of Cu. and 5.9 p.p.m. of Fe. In the hydrogen cycle the cation resins have a greater capacity for removing copper than

in the potassium cycle. In the H-cycle, the pH of the wine dropped from 3.53 to 1.94 when a volume of wine about equal to that of the resin was passed over the resin bed in the course of about 5 minutes, and all the copper but only a small portion of the iron was removed. The resin became saturated when about 13-17 times its volume of wine had passed through and then copper was again found in the wine. At this time the greatest decrease in iron content occurred, and the pH began to rise again. In the K-cycle the pH changes were less but only half the copper was removed during the passage of between 1 and 9 volumes of wine. The K-resin, however, had a much higher exchange capacity for iron than did the H-resin. The final copper content of the wine after the resin had become saturated with the cations in wine was greater than the initial copper content of the wine, indicating concentration of copper in the resin bed. In the H-cycle the wine became sour in flavor while in the K-cycle it became salty. In both cases the wine lost its bouquet and was unpalatable. The anion exchange resins were first converted into tartrates by activation first with ammonium hydroxide and then with potassium acid tartrate. The data obtained for Amberlite IR-4B are shown in Table 4 and Figure 3. The anion exchange resins used apparently had the ability to complex both copper and iron as both were removed from the wine. The anion exchange resin was as efficient in removing iron from wine as the K-cation exchange resin, and more efficient in removing copper. The flavor and color of the wine were affected adversely.

TABLE 2
Effect of calcium phytate on iron and copper content of wine

Time (hours)	Initial Cu content	Final Cu content	Initial Fe content	Final Fe content
	p.p.m.	*p.p.m.*	*p.p.m.*	*p.p.m.*
1	0.6	0.6	8.1	4.2
2	0.6	0.6	8.1	4.0
4	0.6	0.6	8.1	3.6
5	0.6	0.6	8.1	3.9
24	0.6	0.6	8.1	2.6
48	0.6	0.6	8.1	2.5
96	0.6	0.6	8.1	2.5
1	4.8	4.8	8.1	4.4
21	4.8	4.7	8.1	2.4
45	4.8	4.8	8.1	2.2
100	4.8	4.5	8.1	2.6
120	4.8	4.5	8.1	2.4
1	9.2	8.9	8.1	4.0
21	9.2	9.2	8.1	2.4
45	9.2	9.2	8.1	2.5
100	9.2	9.0	8.1	2.4
120	9.2	8.9	8.1	2.3

TABLE 3
Copper and iron removal from white table wine by Dowex-50 resin

Sample	Total volume	H resin			K resin		
	ml.	pH	Cu	Fe	pH	Cu	Fe
Original wine	...	3.53	2.2	5.9	3.60	2.0	6.0
1	25	1.94	0	5.0	3.88	1.0	0.4
2	150	1.94	...	...	3.72	...	...
3	275	1.95	...	...	3.70	1.2	0.7
4	400	1.95	...	...	3.68	...	...
5	525	2.00	0.25	4.25	3.66	1.65	2.2
6	650	2.02	...	...	3.65	...	...
7	775	2.10	0.70	5.03	3.65	1.8	2.0
8	900	2.27	0.95	...	3.65	...	...
9	1025	2.50	1.43	5.2	3.64	2.0	2.6
10	1150	2.72	1.90	...	3.64	...	...
11	1275	2.92	2.97	5.1	3.64	2.0	2.9
12	1400	3.12	3.05	...	3.63	...	...
13	1525	3.28	...	...	...	...	...
14	1650	3.42	3.15	5.03	...	...	...
Average flow rate		5.3 ml./min.			5.55 ml./min.		

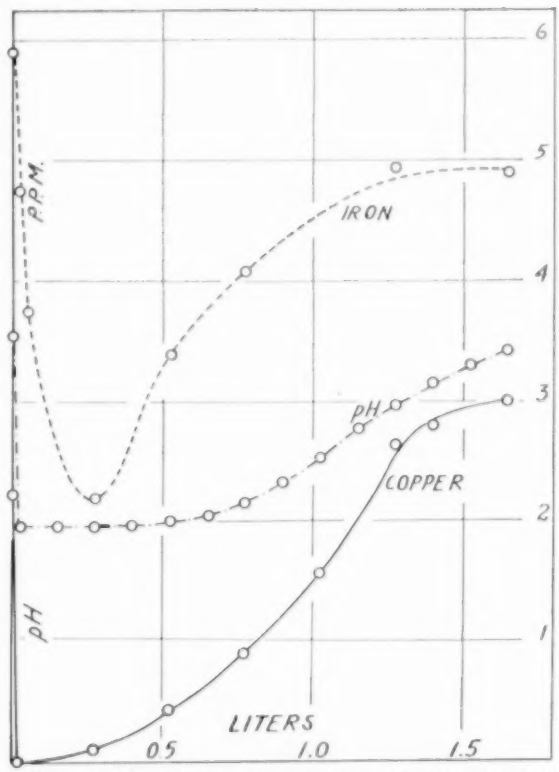

Figure 1. Changes in copper and iron content and pH of wine flowing through 30 ml. (wet volume) of Dowex-50 cation exchange resin at 5 ml. per min. in the H-form.

Austerweil and Pecheur *(4)* found similar decreases in pH value during treatment of wine with cation exchange resins but proposed the treatment with anion exchange resin subsequent to treatment with cation exchange resin to correct this condition.

Rate of Precipitation of Copper and Iron Ferrocyanides

The addition of ferrocyanide to ferric ions results in the production of blue substances generally called Prussian blues. When an excess of ferric ions is present an insoluble Prussian blue forms but the equimolecular quantities of ferric ion and ferrocyanide a colloidal solution forms. The formation of Prussian blue, however, is not simply a mutual precipitation of ferric and ferrocyanide ions and is not an instantaneous reaction, but actually has been found to be a relatively slow reaction, Davidson *(16)*. Davidson *(16)*, for example, points out that when equal volumes of 0.0002 formal $K_4Fe(CN)_6.3H_2O$ and ferric alum, $NH_4FE\ (SO_4)_2.12H_2O$ are mixed the original colorless liquids form a yellow solution which quickly changes to green and finally becomes blue in one minute. If the solutions taken are three times as strong the succession of colors is complete in one second, while if the solutions are only one-third as strong as the original the reaction requires the better part of an hour. The formation of Prussian blue also is known to be hindered by excess of ferric ions and hastened by excess of ferrocyanide. Davidson

TABLE 4
Copper and iron removal by Amberlite IR-4B resin

Sample	Total volume	pH	Cu	Fe
Original wine		3.59	2.15	5.85
1	25	3.99	0.48	0.6
2	150	3.83	0.83	1.0
3	275	3.77	1.05	1.0
4	400	3.74	1.20	2.5
5	525	3.72	1.30	2.7
6	650	3.71	1.42	—
7	775	3.71	1.42	2.5
8	900	3.71	—	—

(16) has explained this paradox by pointing out that, preceding the formation of Prussian blue, the ferric ion is almost completely reduced to the ferrous ion by the ferrocyanide ion which is simultaneously oxidized to ferricyanide ion. If ferric and ferrocyanide ions are actually involved in the formation of Prussian blue, then it is slow because the ions required for it practically disappear when mixed, owing to the redox reaction which they undergo instantaneously. In confirmation of this, Davidson *(17)* showed that mixtures of ferrous ions and ferricyanides behave similarly to those of ferric ions and ferrocyanides. Here an excess of ferrous ions favors formation of a blue product from ferricyanide ion.

Vorländer in 1913 (cited by Davidson *(16)* reported, however, that unlike the formation of a blue from ferric and ferrocyanide ions, which is slow, the production of a blue from ferrous and ferricyanide ions is instantaneous. Davidson *(16)* accounted for the color and composition of soluble (colloidal) Prussian blue by assuming polynuclear complex formation between ferric ion and the nitrogen end of the cyanide groups present

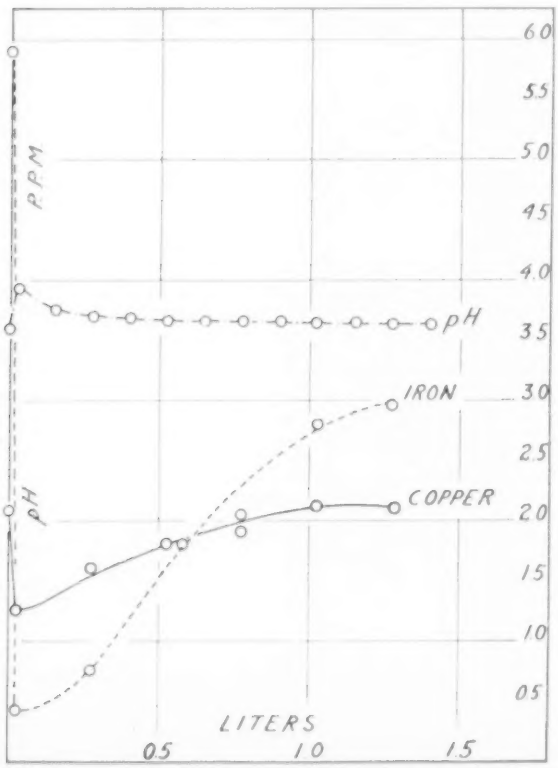

Figure 2. Changes in copper and iron content and pH of wine flowing through 30 ml. (wet volume) of Dowex-50 cation exchange resin at 5 ml. per min. in the K-form.

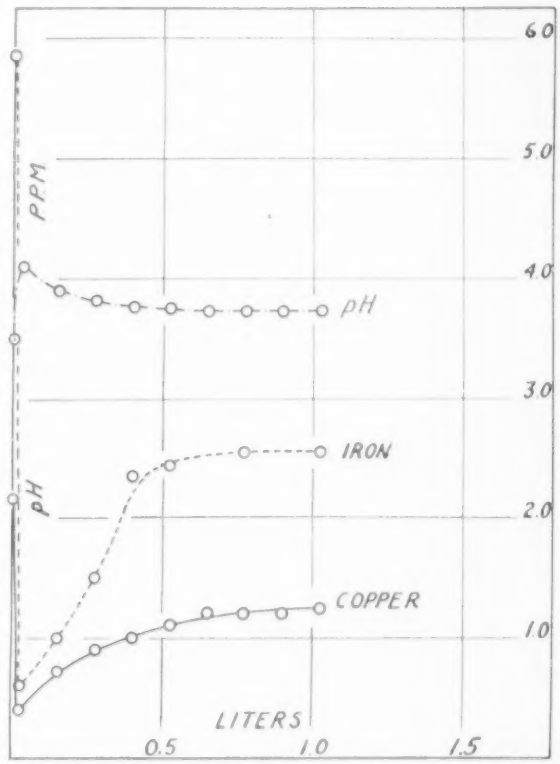

Figure 3. Changes in copper and iron content and pH of wine flowing through 30 ml. (wet volume) of Amberlite 1R-4B anion resin in the tartrate form at the rate of 5 ml. per minute.

in ferrocyanide, and proposed berlinate as a name for the complex of ferric and ferrocyanide ions.

McBain *(33)* in discussing the preparation of colloidal sols explains their formation in accordance with von Weimarn's precipitation laws. Von Weimarn *(57)* assumes that usually the reaction producing insoluble substances is instantaneous and that to form a particle from such molecules it is necessary that the concentration of the reaction product exceeds its true solubility. Each particle grows from an initial nucleus so that in the formation of colloidal sols one must consider, besides the stabilizing agent, the rate of formation and number of nuclei present and the rate of growth of nuclei. Von Weimarn *(57)* showed that particles will be at their finest when formed from very dilute or from very concentrated solutions. In very dilute solutions diffusion is very slow because the concentration gradient is so small, and sufficient nuclei will usually appear to relieve the slight supersaturation locally. In extremely concentrated solutions nuclei appear so quickly and in such numbers that supersaturation is immediately relieved before appreciable diffusion can occur. McBain illustrates these effects by pointing out that if very dilute solutions of ferric chloride and potassium ferrocyanide are mixed there is formed a transparent sol of Prussian blue which passes unaltered through filter paper. At a more concentrated range a precipitate of Prussian blue is formed a few moments after mixing which is completely retained on the filter paper. When one part of a saturated solution of ferric chloride is poured into the chemically equivalent amount of satu-

rated potassium ferrocyanide (two parts) solution a thick cheesy paste results.

Both the colloidal aspects and the oxidation-reduction equilibrium must be considered in developing the optimum conditions for heavy metal removal by ferrocyanide. The conditions must be such that a readily filterable insoluble ferrocyanide precipitate rather than a colloidal sol is formed. Empirically it has been found in the industry that the quantity of potassium ferrocyanide required must be added in a fairly dilute but not too dilute solution, and introduced into the wine so that it is thoroughly and completely dispersed throughout to avoid local supersaturation, that aeration during fining should be avoided and that sufficient time should be allowed for precipitation of the heavy metal ferrocyanides before filtration. Additional fining agents, gelatin tannin, casein or bentonite, if used, should be added after the introduction of potassium ferrocyanide and initial formation of particles of metal ferrocyanides.

Procedure. To obtain data on the rate of formation and precipitation of copper and iron ferrocyanides, slightly cloudy samples of French Colombard wine adjusted to 8.1 p.p.m. of iron and 0.6 to 10 p.p.m. of copper were mixed with potassium ferrocyanide in amounts equivalent to the metal ions present and twice the equivalent amount by bubbling in nitrogen gas. The rate of formation and settling of the ferrocyanide was followed by periodically measuring transmission at 660 mμ in a Klett-Summerson colorimeter. This procedure was also followed in samples of the same wine after clarification with bentonite and aeration by filtration and in deaerated oxygen free samples.

Typical of the data obtained are those shown in Figure 4 for clarified, deaerated wine adjusted to varying levels of copper content and treated with one equivalent of ferrocyanide. It is seen that the optical density first increases rapidly as particles of metal ferrocyanide form and then decreases as these grow in size and precipitate. The time required to reach maximum cloudiness following addition of ferrocyanide varied from 45 minutes for wine containing 0.6 p.p.m. of copper to 25 minutes for that containing 10 p.p.m. of copper. The formation of maximum cloudiness was longer in clarified and aerated wine, somewhat shorter in deaerated wine and shortest in the cloudy wines. Difference in concentration of copper had more effect than difference in treatment of wine. Doubling the concentration of ferrocyanide added had little or no effect on rate of precipitation. In all cases the copper was more rapidly and effectively removed than was the iron.

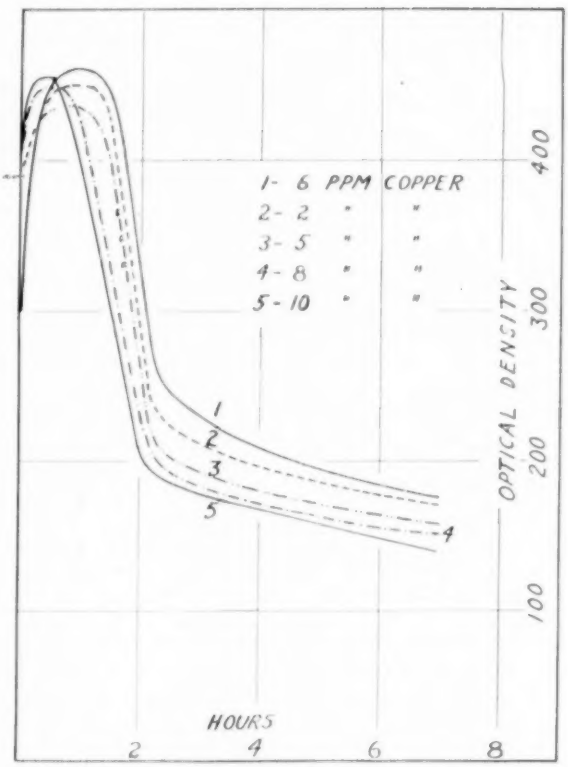

Figure 4. Rate of formation and precipitation of copper and iron ferrocyanides in clarified deaerated wine containing 5.8 p.p.m. of iron and adjusted to various levels of copper content after addition of one equivalent of potassium ferrocyanide. Ordinates are Klett-Summerson colorimeter readings using a 660 mμ color filter.

Rate of Formation and Precipitation of Copper Rubeanates

The rate of formation and precipitation of copper in 12 types of wine, a red wine vinegar and a white wine vinegar at various levels of copper following the addition of rubeanic acid at various stoichiometrical levels (equivalent, half equivalent and two equivalent amounts) was investigated by a procedure similar to that described in the ferrocyanide tests. The time required to attain maximum cloudiness varied with the type of wine, the initial copper concentration and the equivalents of rubeanic acid added. The data obtained for French Colombard wine, adjusted to from 0.2 to 10 p.p.m. of copper, to which one equivalent of rubeanic acid was added are shown in Figure 5. With this wine no change in optical density was observed at 0.2 and 1 p.p.m. of copper, maximum clouding occurred at 20 hours at 2 p.p.m. and at 5 hours for 4 to 10 p.p.m. As in the case of ferrocyanide fining, following the peak of maximum optical density there was a rapid decrease owing to flocculation and precipitation of the copper rubeanate.

Sequestering of Copper and Iron by Versene

The equilibrium constant for the Versene chelate compounds at 20° C. given in Anon. *(3)* is 3.89×10^{10} for Ca^{++}, 3.01×10^{14} for Fe^{++}, 2.51×10^{25} for Fe^{+++}, and 6.02×10^{18} for Cu^{++} for the reaction: $Me^{n} + Ver^{4} \rightleftarrows MeVer^{n-4}$. The order of chelation of these metals by Versene should be $Fe^{+++} > Cu^{++} > Fe^{++} > Ca^{++}$ if only the metal ion and the Versene were involved. In a product like wine where the existence of metals in complexes with organic acids as well as the free ions would be expected, the pH and composition would affect the sequestering power of Versene added. Krum and Fellers *(28)* reported on the clarification of wines containing both iron and copper induced cloudiness following addition of equivalent amounts of sodium ethylene diamine tetroacetate distributed under the trade name of "Sequestrene." Their data indicates that addition of 8 parts of "Sequestrene" per part of metal present produced clarification but does not indicate whether or not the clarification of copper casse was caused by added Versene or exposure to air during observation. As is shown in Table 1, wines to which "Versene" was added

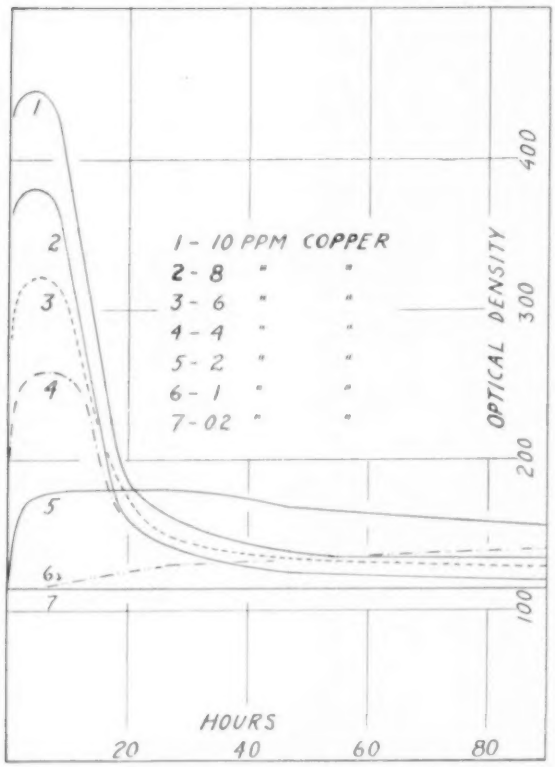

Figure 5. Rate of formation and precipitation of copper rubeanate from wine adjusted to varying levels of copper content after addition of on equivalent of rubeanic acid. Ordinates are Klett-Summerson colorimeter readings using a 660 mμ color filter.

apparently do not bind copper as firmly as it is bound by the diethyl dithio carbamate used in the colorimetric determination but there is some eveidence that the iron is firmly chelated so that it will no longer react either with added ferrocyanide or thiocyanate. Wines suspected of containing added Versene have been found to continue to form copper casse and no longer respond to ferrocyanide fining *(19)*.

Procedure. In order to determine whether Versene by chelating copper or iron could prevent formation of metallic hazes and deposits several white wines were adjusted with added copper and iron, phosphate and tannate and stored under conditions favorable to formation of blue ferric tannate, the white ferric phosphate and the white "cuprous sulfide" casse. A white pinot, two Sauterne wines (dry and sweet) and a Rhine wine were adjusted to 1 and 5 p.p.m. of Cu, to 5 and 10 p.p.m. of iron, with and without added Versene (1 and 5 equivalents), filled hot and cold into crown capped bottles and stored in completely filled bottles in light to promote copper casse formation. For the ferric tannate tests samples of the above wines were adjusted to 0.125 g. tannic acid per 100 ml. and stored under oxidizing conditions in partly filled cork stoppered bottles. For the ferric phosphate tests, samples of the above wines containing added 0.1% of dibasic sodium phosphate were stored under oxidizing conditions.

Within two weeks the ferric phosphate and ferric tannate samples without added Versene were cloudy and the cloudiness increased on storage. Those containing added Versene either did not cloud or did not cloud as much, indicating suppression of ferric casse by Versene added in equivalent amount. In the copper casse samples the wines which were heated to 140° F. (60° C.) and filled hot did not show any casse after storage for a month either with or without Versene. Unheated wines showed more tendency to clouding than heated ones. These investigations are being continued. Definite proof that Versene inhibited copper casse formation was not obtained as the cloud and precipitate in the test samples was not analyzed for copper or exposed to air. In initial stages of copper casse formation mere exposure to air is sufficient to dissolve the insoluble copper deposits formed. The indications are that Versene is more effective in inhibiting ferric casse than copper casse.

Toxicology of Ferrocyanide and Possible Substitutes

As pointed out previously a suitable substitute for ferrocyanide must be relatively less toxic and should not introduce residues into the wine. It was for the latter reason that treatment of wine with chelating or sequestering agents which would form stable soluble complexes in the wine was not considered as desirable as the use of one which would form insoluble complexes. Apart from possible toxicity there is also the problem of meeting the Alcohol and Tobacco Tax Unit and the Food and Drug Administration rulings and restrictions on such residues. Versene, which was available to us at the start of our work early in 1948, was not considered desirable for these reasons. The relative toxicity of the ferrocyanides and their tendency to hydrolyze into the toxic free cyanide has not been established completely. In the older literature Prussian blue is referred to as "the well-known non-poisonous pigment." One of the most thorough studies on the pharmacology of ferrocyanide was that of Miller and Winkler *(35)* on the clearance of sodium ferrocyanide in man. They found that it caused renal irritations, and when injected at the rate of 20 mg. per kg. of body weight produced definite nephritic symptoms. Ethylene diamine tetraacetate (Versene), manufactured under the F. C. Bersworth patents, was reported not to be toxic in dosages given to humans up to 5 g. over an 8-hour period. At these levels and rates of administration hypocalcium tetany did not occur *(3)*. Krum and Fellers *(28)* conducted acute and chronic toxicity tests with rats and found no toxic effects either with single oral doses of 4 g. per kg. body weight or on long time feeding of basal ration plus 0.5% of Sequestrene. Acute and chronic toxicity tests with rubeanic acid are being carried out. The present indications are that it is not toxic at the levels used, but in the possible industrial application of rubeanic acid for copper removal there are, of course, additional considerations.[4]

SUMMARY AND CONCLUSIONS

While the factors influencing the tolerance of wine to heavy metals, particularly copper are known in general, our ability to predict the stability of a particular wine

[4] A first consideration is cost; at the present time the Organic Chemical Sales Division of Eastman Kodak Company lists rubeanic acid (dithioxamide) at $3.00 per 10 grams in comparison with potassium ferrocyanide at $0.21 per pound in barrel lots. This price differential, of course, can be reduced as demand increases. Another consideration is the need to dissolve rubeanic acid in alcohol to avoid hydrolysis so that small amounts of alcohol would be added to the wine with it. Its advantage is that unlike Versene and related compounds its use will not result in the presence of residues in the wine.

under particular storage and distribution conditions is still incomplete. More information on the chemical and colloidal changes occurring during copper and iron casse formation is needed.

The possible substitutes for the removal of copper, which presents the most important problem today, were discussed. Removal of copper by precipitation with relatively non-toxic organic compounds was investigated. Of the compounds tested, rubeanic acid was most promising. Calcium phytate was not found to precipitate copper under the conditions used and produced only partial reduction in iron content.

The conditions of precipitation of copper and iron as insoluble ferrocyanides were investigated and an adsorption compound of potassium ferrocyanide with Prussian blue, with or without addition of denatured proteins was suggested as a possible substitute for potassium ferrocyanide.

The rate of formation of copper rubeanates and of copper and iron ferrocyanides in wine was studied and some of the factors influencing formation of particles, their growth and precipitation were discussed.

The removal of copper and iron from wine by the use of both cation and anion exchange resins was investigated. The adverse effect of cation exchange resins tried in the H-cycle on flavor ruled out their use. In the K-cycle their exchange ability for copper was reduced and they also affected the flavor of wine adversely.

Acknowledgments

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Studies on the Processing of Red Cherries. I. Changes in Fresh Red Cherries Caused by Bruising, Cooling, and Soaking

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Data are given that show how the weight, firmness, and soluble-solids content of fresh red cherries are affected by bruising, temperature, soaking, and period of storage. Not all changes that occur in cherries when soaked are caused by soaking alone. Bruising lessens the gain in weight of cherries when soaked and increases the loss of soluble solids and acids.

Soaking fresh red cherries in cool water before processing provides a convenient means of cleaning, cooling, and storing them. Some changes that occur in the cherries when they are soaked have been described previously *(2, 3, 4, 5, 6)*. No general statement concerning the changes can be made, however, since the studies were somewhat limited in scope and conflicting results were obtained. Peterson *(4)* found that cherries lose weight during the first 5 or 6 hours of soaking, but thereafter steadily gain weight. The cherries used by Swingle *(6)* decreased in weight as much as 4.4% when soaked for 4 hours. Langer and Fisher *(2)*, however, observed no significant changes in the weight of cherries when soaked. Reynolds and Reynolds *(5)* expressed the opinion, commonly accepted in the industry, that cherries retain all their natural juices during soaking. On the other hand, Peterson *(4)* and Marshall *et al. (3)* reported that cherries lose appreciable quantities of soluble solids if soaked for more than about 12 hours.

Part I of the present study shows how conflicting results on the effects of soaking might easily be obtained. Cherries from the same lot, for instance, were made to gain, lose, or show no change in weight merely by varying the degree of bruising. The cherries were affected also by factors such as the temperature and duration of soaking and the period of natural aging or "conditioning." The present paper deals only with changes in fresh cherries; changes in canned cherries induced by various preprocessing treatments are described in the second part of this study *(1)*.

MATERIALS AND METHODS

In most experiments, commercial cherries were not used because they were not sufficiently uniform in size, maturity, extent of bruising and post-harvest history. Instead, the cherries (Montmorency) were obtained from 2 similar trees in the orchard at Michigan State College, East Lansing, Michigan, in 1949. The trees had been sprayed with Tennessee 26, a copper fungicide. The cherries were picked with a minimum of bruising, and all samples were essentially uniform in respect to maturity and size of cherries. Only samples of average maturity, as indicated by their red color, were used.

Experiments were begun within 2 hours after the cherries were picked, and were conducted in rooms of constant temperature. Firmness was measured with a pressure tester *(8)*, and the soluble-solids content was estimated with an Abbé refractometer, except where otherwise noted. Some samples were bruised by dropping the cherries from a predetermined height onto a stainless steel tray.

RESULTS

Effects of soaking. Unbruised cherries soaked in distilled water for 77 hours increased 10% in weight (Table 1). The increase was more rapid at 21° C. (70° F.) than at 10 or 1° C. (50 or 34° F.). At the last 2 temperatures, the increase was

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roughly proportional to the soaking period, the increase being 4% after 24 hours. With a gain in weight of 10%, a large proportion of the cherries cracked.

The firmness of the cherries increased with prolonged soaking at each of the 3 temperatures. The increase was greatest and most rapid in cherries soaked at 1° C. (34° F.). The increase, however, cannot be attributed entirely to soaking, as will be shown. Cherries soaked in a dilute solution of calcium chloride were slightly firmer than those soaked in water. Meanwhile, the soluble solids content of all samples decreased, the largest decrease occurring in the samples soaked at 21° C. (70° F.). Table 3 and the results of Peterson *(4)* show also that cherries decrease in acidity when soaked.

In some respects the behavior of bruised cherries (dropped twice from a height of 3 feet) contrasted sharply with that of unbruised fruit. At 21° C. (70° F.) bruised cherries lost weight when soaked, and at 1° C. (34° F.) they had gained only 1% in weight after 77 hours. Although they increased in firmness during soaking, their firmness at all stages was lower than that of unbruised cherries. Furthermore, although the changes in their soluble-solids content paralleled those of unbruised cherries, the total quantity of soluble solids leached from them was about double that from unbruised fruit.

The effects of bruising on soaked cherries were roughly proportional to the extent of bruising (Table 2). Slightly bruised cherries (dropped from a height of 1 or 2 feet) responded to soaking in a manner not much different from that of unbruised cherries. The differences became pronounced, however, when the cherries were moderately to severely bruised (cherries dropped 2 to 4 times from a height of 3 feet).

Limited data (bottom of Table 2) indicated that many samples of commercial cherries were moderately to severely bruised, since they had a comparatively low degree of firmness and did not gain appreciably in weight when soaked. A satisfactory evaluation of commercial cherries, however, must await further study; the effects of bruising may be altered or obscured by differences in their post-harvest history.

TABLE 2

Some effects of bruising red cherries

Cherries and treatment[a]	Firmness before soaking, compression %	Change in weight during soaking %	Loss of soluble solids during soaking[b] %
Experimental cherries			
Control, unbruised	24	+3.8	2.9
Dropped 1 ft.		+3.0	4.0
Dropped 2 ft.	25	+2.3	4.6
Dropped 3 ft.	27	+1.7	5.5
Dropped 3 ft. 2 times	32	+0.2	6.4
Dropped 3 ft. 3 times	37	−0.5	8.6
Dropped 3 ft. 4 times	42	−0.6	14.1
Commercial cherries			
1. No treatment	24		
2. No treatment	26	+0.4	
3. No treatment	26	−0.8	
4. No treatment	27	+1.5	
5. No treatment	28		
6. No treatment	29	+1.3	
7. No treatment	29	+1.1	
8. No treatment	33		

[a] All samples were soaked in tap water at 10° C. for 23 hours.

[b] Soluble solids leached from the cherries into the soaking medium were determined gravimetrically.

TABLE 3

Effects of bruising and removal of stems on the weight and loss of soluble solids and acids from soaked cherries

Cherries and treatment[a]	Change in weight, %				Loss of soluble solids, %[b]				Loss of acids, %[c]
	Hours soaked				Hours soaked				Hours soaked
	2	7	16	24	2	7	16	24	24
Stems attached	+0.2	+0.8	+1.7	+2.4	0.1	0.1	0.2	0.3	0.6
Stems removed	+1.5	+3.1	+5.4	+7.1	0.7	1.2	2.3	3.1	1.2
Stems removed and cherries bruised	−1.1	−0.7	0.0	+1.0	2.9	4.4	6.5	8.4	10.5

[a] Cherries were soaked in tap water at 10° C. The bruised cherries were dropped twice from a height of 3 feet.

[b] Soluble solids leached from the cherries were determined gravimetrically.

[c] Total acids leached from the cherries were determined by titration and calculated as malic acid.

The principal pathway for the exchange of substances between the cherries and the soaking medium was the area of tissue exposed by the removal of stems (Table 3). Cherries with stems attached gained 2.4% in weight and lost only 0.3% of their soluble solids and 0.6% of their total acids when soaked for 24 hours. The corresponding values for cherries without stems were 7.1, 3.1, and 1.2%. The exchange of materials between the bruised cherries and the soaking medium also probably took place largely through the tissues exposed by the removal of stems, since there were no lesions in the skins. The effect on the exchange of permitting cherries to stand in air for several hours at room temperature after removal of their stems was not determined.

Effects of storage in air. To help define the changes in cherries caused by soaking alone, control samples were stored in air for similar periods and at the same temperatures as the soaked cherries. Since fresh cherries are alive and capable of changing within themselves, not all the changes that occur during soaking can be ascribed to soaking.

Lowering the temperature of the cherries caused a rapid increase in firmness (Table 4). The difference in firmness among samples stored at 21, 10, and 1° C. (70, 50, and 34° F.) was maintained or became greater throughout the experiment. There was, however, a second factor, not completely dependent on temperature, which affected firmness. This factor included the vital changes in the cherries themselves. It was expressed by the gradual increase in firmness of cherries stored at a constant temperature of 1° C. It also was shown by the initial increase in firmness of cherries picked and stored at 21° C. The cherries were least firm immediately after being picked, owing partly to the inevitable bruising attendant on the removal of their stems. After a short period of aging or "conditioning," however, the bruised areas evidently were repaired or strengthened, and the cherries temporarily became firmer. Similar increases in firmness of freshly picked cherries were observed in numerous other samples.

During storage, the cherries of course lost weight, but at the same time the percentage of soluble solids increased. The greatest changes occurred in the samples stored at 21° C. At 1° C. the cherries were relatively stable. Some unbruised sam-

TABLE 1

Changes in unbruised and bruised red cherries during soaking in distilled water or in 0.1% calcium chloride solution

Temperature and treatment[b] of cherries	Change in weight, %			Firmness, % compression				Soluble solids content, %				Loss of soluble solids, % of total[c]
	Hours soaked			Hours soaked				Hours soaked				Hours soaked
	10	24	77	0	10	24	77	0	10	24	77	77
21° C.												
Unbruised	+3	+6	+10	24	23	23	18	13.0	12.7	11.8	10.0	7
Bruised	−1	−1	−1	33	32	30	25	13.0	12.8	12.4	10.2	14
10° C.												
Unbruised	+2	+4	+10			22	17	13.0	12.6	12.0	10.5	7
Unbruised, 0.1% CaCl	+2	+4	+10			21	15	13.0	12.6	12.6	11.0	7
1° C.												
Unbruised	+2	+4	+10		21	20	16	13.0	12.8	12.7	10.9	5
Bruised	0	0	+1		27	26	23	13.0	12.8	12.7	11.1	12

[b] The bruised cherries were dropped twice from a height of 3 feet.

[c] Soluble solids leached from the cherries into the soaking media were determined gravimetrically.

TABLE 4

Changes in red cherries during storage in air

Temperature[1] and treatment[2] of cherries	Loss in weight, %					Firmness, % compression						Soluble-solids content, %			
	Hours stored					Hours stored						Hours stored			
	2	8	26	51	100	0	2	8	26	51	100	0	26	51	100
21° C.															
Unbruised	1	1	3	5	10	24	23	22	23	24	25	13.9	14.3	14.8	14.9
Bruised	1	2	5	8	13	—	30	31	29	29	29	13.9	14.2	14.6	15.1
10° C.															
Unbruised	0	0	1	1	2	[3]	22	22	23	23	21	13.9	14.5	14.1	14.5
1° C.															
Unbruised	0	0	1	1	1	[3]	21	21	21	20	19	13.9	13.9	14.1	14.3
Bruised	1	1	1	2	2	[3]	27	26	25	23	23	13.9	14.6	14.0	14.2

[1] The relative humidities at 21, 10, and 1° C. were 56, 73, and 81%, respectively.
[2] The bruised cherries were dropped twice from a height of 3 feet.
[3] Initial readings were not made because the cherries had not cooled to the temperatures indicated.

ples were stored in air at this temperature for as long as 12 days without spoilage.

Bruised cherries lost more weight and were less firm than were unbruised cherries. After about 51 hours' storage, however, bruised cherries at 1° C. were firmer than were unbruised cherries at 21° C.

DISCUSSION

From the collective data in the tables, a number of observations may be made concerning the changes in firmness in the stored and soaked cherries. The firmness of both bruised and unbruised fruit was affected by at least four factors: a, temperature, b, soaking in water, c, treatment with calcium ions, and d, natural aging or "conditioning" of tissues. The increase in firmness caused by lowering the temperature several degrees was immediate and significant. On the other hand, an increase in firmness due to soaking was evident only after the cherries had been soaked for a relatively long period. Cherries soaked for 24 hours were not significantly firmer than similar cherries stored in air. It is possible, however, that somewhat different results may be obtained with commercially handled cherries. The role of calcium ions in enhancing the firmness of tissues is well known. Changes in firmness associated with physiological aging depended partly on the conditions of storage of the cherries. With prolonged storage at 1° C. (34° F.) there was an increase in firmness of both bruised and unbruised cherries.

Soaking the cherries induced several changes that would not have occurred otherwise. Among these were the leaching of soluble solids and acids from the cherries into the soaking medium, a decrease in percentage of soluble solids, an increase in firmness beyond that attributable to aging or to a lowering of temperature, and, in the case of unbruised cherries, an increase in weight and in the number of cherries which cracked.

The present findings apply only to freshly picked, raw cherries treated under carefully controlled conditions. Similar results were obtained with many different lots of cherries during 1950 and 1951. Application of the findings to commercially handled cherries, however, may be rendered difficult by many variable and uncontrolled factors which act simultaneously upon the cherries. Among these are extent of bruising, recurrence of bruising, temperature and time interval between bruising and processing, and temperature and time interval between picking and processing.

The yield and quality of heat-processed cherries are not always predictable from the weight and quality of raw fruit *(1, 7)*. Gain in weight associated with soaking unbruised cherries, for instance, does not necessarily mean increased profits to the processor, since much of the gain is lost in the subsequent processing steps. Additional studies will be made on the relation between post-harvest treatments of fresh cherries and the resulting yield and quality of the processed product.

SUMMARY

Unbruised red cherries soaked in water increased in weight and firmness, and decreased in soluble-solids content and acidity. Bruised cherries, however, did not gain significantly in weight and lost appreciable quantities of soluble solids and acids in the soaking medium. The effects of bruising were proportional to degree of bruising. Most commercial cherries are rather severely bruised.

The principal pathway for the exchange of substances between the cherries and the soaking medium was the area of tissue exposed by removal of stems. Both bruised and unbruised cherries became firmer and lost less soluble solids when soaked at 1° C. (34° F.) than when soaked at 10 or 21° C. (50 or 70° F.). Not all the changes that occurred in the cherries during soaking were caused by soaking alone. Cherries stored in air at 1° C. increased steadily in firmness and in percentage of soluble solids. Unbruised cherries kept well under these conditions for as long as 12 days. The firmness of both bruised and unbruised cherries was increased by lowering the temperature, by treating them with calcium ions, by permitting them to age or "condition" naturally, and by soaking them in water.

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Studies on the Processing of Red Cherries. II. Some Effects of Bruising on the Yield and Quality of Canned Montmorency Cherries[a]

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The bruising of fresh cherries prior to the soaking period increased both the ratio of drained weight to put-in weight and the tenderometer reading of the canned product. Under the experimental conditions employed, however, bruising had almost no effect on final yield, color, and soluble-solids content. Cherries processed immediately after being picked were comparatively soft and gave a low yield. Storing the cherries either in air or in water for 15 hours at 65° F. increased both their firmness and yield.

Although the procedure of soaking red tart cherries in cold water prior to processing has been in general use for more than 30 years, published information on the effects of this treatment on the subsequent behavior of cherries during processing is relatively scarce. It is commonly believed that soaked cherries may be pitted with less waste and that the pitted fruit "bleeds" less than does fruit not soaked *(5)*. Reynolds and Reynolds *(6)* expressed the opinion that cherries soaked in cold water retain all their natural juices, become firm, and hold their shape well in canning.

Griswold *(1)* found, however, that soaking cherries in cold water caused deterioration in the color of the canned product. The deterioration was proportional to period of soaking. Similar results were obtained by Marshall and co-workers *(4)* who observed also that soaking caused a loss of soluble solids and concluded that soaking was not essential for preventing excessive pitting losses.

Some of the changes that cherries undergo when soaked in cold water have been described previously *(3, 4, 5, 10)*. Whittenberger and Hills *(10)* observed that the changes were dependent in part on the degree of bruising. Bruised cherries did not gain significantly in weight and lost appreciable quantities of soluble solids into the soaking medium. By contrast, unbruised cherries gained steadily in weight and lost only small amounts of soluble solids. Since most lots of commercially handled cherries are rather severely bruised by the time they arrive at the processing plant, the results of this study on bruising should be of interest to cherry processors.

The present paper, an extension of the previous study *(10)*, discusses the effects of bruising and of various cooling and soaking treatments on the pitting losses, yield, and quality of canned cherries.

[a] Report of a study made under the Research and Marketing Act of 1946. Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Michigan, June 9, 1952.

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MATERIALS AND METHODS

Montmorency cherries were obtained from the Michigan State College orchard at East Lansing and hauled to the fruit-processing laboratory of the Department of Horticulture. The fruit was obtained from 20-year-old trees which had been sprayed with a proprietary copper fungicide, Tennessee 26. The cherries were picked without stems and handled carefully to avoid crushing or bruising. The fruit was picked in the morning and immediately transported to the laboratory, where it was sorted to remove all defective and bruised cherries. Bruised fruit was detected by gently rolling each cherry with one's fingers and noting the lack of turgidity.

In all experiments, the cherries were sorted for maturity on the basis of color. At the harvest stage, most of the cherries were a bright red, but a large number had an orange-red color; some had a dark-mahogany shade. Fruit corresponding to these three color groups was arbitrarily called ripe, under-ripe, and over-ripe, respectively.

Some lots of cherries were bruised by a standard procedure. Each cherry was dropped twice from a height of 3 feet onto a 16-gauge stainless steel pan. Care was taken to avoid dropping the cherries onto other cherries, because this produced less bruising and made for non-uniformity of samples. This extent of bruising seemed equivalent to that observed in many lots of commercially harvested fruit. The bruised cherries were held in trays for 2 hours before soaking or cooling. This allowed some of the juice to exude onto the surface, as it commonly does in commercially handled fruit.

Approximately 2-kg. lots of cherries were weighed into cylindrical wire mesh baskets, 6 inches deep and 10 inches in diameter, and soaked in 6 liters of distilled water in a 12-inch diameter glass cylinder held in a room at 50° F. (10° C.). By starting with water at 42° F. (5.5° C.) the cherries were cooled from about 75 to 50° F. (23.9 to 10° C.) within a few minutes. The cherries were weighed at intervals during soaking. Weighings were made after removing the baskets from the water and allowing the fruit to drain for 5 minutes. The initial weighing was always made after soaking for 10 minutes. This 10-minute soak, or rinse, removed the surface juices and gave the correct net weight for the wet fruit, which could be used as the starting point for determining gain or loss on soaking. Soluble-solids determinations were made on aliquots of the soaking solution by evaporating to dryness.

The cherries were pitted in the same pilot-scale Dunkley pitter used by Marshall *et al. (4)*. Loss in weight due to removal of pits and loss of juice was recorded for each lot. The pitted cherries were canned in No. 2 cherry enamel cans. The "put-in" weight was 444 g. of fruit per can. The cans were exhausted to a center temperature of 165-170° F. (74-76.7° C.), sealed, processed in boiling water for 12 minutes, cooled and stored at 50° F. (10° C.).

After 4 months' storage, the cans were opened, and the cherries were examined for color, soluble solids, texture, and drained weight. The cans were removed from storage one day prior to examination and allowed to warm to room temperature. The color of the supernatant juice, filtered through No. 1 Whatman paper and diluted 1:5 with distilled water, was evaluated with a Coleman spectrophotometer, Model 14, set at 440 mμ. Preliminary tests indicated that the red pigment was distributed uniformly between the canned cherries and the surrounding liquid.

The soluble-solids content of juice from representative samples of cherries was determined by an Abbé refractometer, and the values were corrected to 68° F. (20° C.) *(2)*. Drained weight was determined by the standard procedure *(7)* used by the U. S. Department of Agriculture, Production and Marketing Administration. Texture was measured on a 100-g. sample of drained fruit, using scale one on the tenderometer.

RESULTS

Effects of bruising. Table 1 shows in detail the various sources of gain or loss during soaking, pitting, and canning. The data are averages of 11 experiments with cherries treated under similar conditions. In all calculations, the percentages are based on the weight of raw, untreated fruit.

TABLE 1

Effect of bruising on the behavior of cherries during processing[a]

Factor Observed	Unbruised	Bruised
	%	%
Weight of raw, dry cherries	100.0	100.0
Weight after rinse at 50° F.	102.8	102.0
Weight after soaking	107.0	103.0
Gain during soaking	4.2	1.0
Pitting loss, total	17.9	16.3
Pit loss	7.3	7.6
Juice loss	10.6	8.7
Weight of pitted cherries	89.1	86.7
Drained weight/put-in weight	82.5	84.7
Yield of canned cherries (drained weight basis)	73.5	73.4
Loss of soluble solids in rinse water	0.4[b]	1.2[b]
Loss of soluble solids during soaking	2.8[b]	4.7[b]
Total loss of soluble solids prior to pitting	3.2[b]	5.9[b]

[a] The data are averages of 11 experiments with cherries bruised before being soaked for 22 hours at 50° F.

[b] Percentage of the total soluble solids in the original cherries.

When rinsed in cold water, unbruised cherries picked up 2.8% of surface water, whereas the bruised cherries gained 2.0%. This difference represents juice which had exuded from the bruised fruit. Since these cherries were already partly wetted, they gained only an additional 2.0% of surface liquid. Analysis of a portion of the rinse water from the bruised cherries showed that 1.2% of the total soluble solids was on the surface. After soaking in water at 50° F. (10° C.) for 22 hours, the unbruised and bruised cherries gained in weight an additional 4.2 and 1.0%, respectively.

The pitting losses are divided into two categories: loss of pits and loss of juice. In addition to the juice lost during pitting, an amount equal to about 5% of the weight of the fruit drains out of the tissue within a few minutes after pitting. No further drainage occurs except under certain conditions of bruising, in which case the pitted cherries may "bleed" for several hours. Juice losses from these various sources are combined in the present text and are discussed under only one heading.

The difference in weight between the unbruised and bruised cherries was greatest at the end of the soaking period. This difference tended to diminish during pitting and canning. The pitting losses were greater for the unbruised fruit (17.9%) than for the bruised fruit (16.3%). Although slightly more flesh adhered to the pits of the bruised cherries, the unbruised cherries lost a significantly greater quantity of juice, which, of course, contained soluble solids. Nevertheless, the quantity of freshly pitted fruit favored the unbruised cherries by the margin of 89.1 to 86.7%.

The weight of the unbruised cherries was affected more by heat processing (canning) than was that of the bruised fruit. To illustrate, the ratio of drained weight to put-in weight of the unbruised cherries was 0.825, whereas that of the bruised cherries was 0.847. The over-all yields, however, from the unbruised and bruised cherries were nearly identical. The yields, on a drained weight basis, were obtained by multiplying the weights of freshly pitted fruit by the ratios of drained weight to put-in weight. The ratios alone did not show accurately the final yields (based on the weights of the original fresh, whole cherries).

Soaking caused changes in the soluble-solids content of the cherries by 2 separate processes. In the first place, soluble solids were leached from the cherries into the soaking medium. The unbruised and bruised cherries lost 3.2 and 5.9%, respectively, of their total soluble solids by this process. These losses, of course, decreased the concentration of soluble solids in the cherries. In the second place, the concentration of soluble solids in the tissues decreased because of dilution with water. For instance, the soluble solids in the unbruised fruit was diluted by about 4.2% and that of the bruised fruit by about 1.0% by the uptake of water during 22 hours of soaking. On a concentration basis, therefore, the soluble-solids contents of the unbruised and bruised cherries were not much different. The bruised cherries showed the greater decrease due to leaching, but the unbruised cherries decreased more by dilution. From a practical standpoint, this means that the unbruised cherries retained nearly all their original soluble solids, whereas the bruised fruit lost appreciable quantities of soluble solids by diffusion; this loss was not recoverable.

Table 2 gives data on the quality of unbruised and bruised cherries after canning. In the lower half of the table, the data are recalculated to the basis of equal drained weights. In commercial practice, the put-in weight usually is adjusted to give the drained weight (13.5 oz. for No. 2 cans) recommended for U. S. Grade A. To attain this drained weight, 16.36 ounces of freshly pitted unbruised cherries would be required, whereas only 15.94 ounces of the bruised cherries would be needed. The increase in tenderometer reading (from 21.5 to 25.2) caused by bruising probably represents an improvement in quality, since many canned cherries are too soft. The color of the processed product, as indicated by the percent transmittance of the juice, was not significantly changed by bruising the raw cherries. A slight over-all loss of soluble solids was associated with bruising.

Effects of maturity and bruising. The effect of maturity on the behavior of unbruised and bruised cherries was studied, following the separation of one lot of cherries into three maturity groups. The cherries were picked from the same tree within a period of 2 hours, and the separation was made on the basis of color. Each of the 3 groups was divided into 2 samples, one of which was purposely bruised and the other left unbruised. These 6 samples were soaked in water at 50° F. (10° C.) for 23 hours before being pitted and canned.

TABLE 2

Effect of bruising on the quality of canned cherries[a]

Factor Observed	Unbruised	Bruised
Put-in weight, ozs.	15.63	15.63
Drained weight, ozs.	12.89	13.24
Ratio of drained weight to put-in weight	.825	.847
Tenderometer reading, lbs. per sq. in.	21.5	25.2
Color, transmittance at 440 mμ, %	40.4	39.5
Soluble solids, %	11.2	10.9
Data above corrected to 13.5 ozs. drained weight		
Calculated put-in weight required, ozs.	16.36	15.94
Color, transmittance at 440 mμ, %	38.8	38.8
Soluble solids, %	11.7	11.1

[a] The data are averages of 11 experiments with cherries bruised before being soaked for 22 hours at 50° F. Cans were stored for 4 months at 50° F.

The soluble-solids contents of the fresh unbruised cherries were 12.1, 14.5, and 15.3% for the immature, ripe, and over-ripe groups, respectively (Table 3). During soaking, the over-ripe cherries gained the most weight (4.4%), and the corresponding immature cherries gained the least (3.7%). In the processed cherries, however, differences in drained weight associated with maturity were small and probably insignificant. The ratios of drained weight to put-in weight were 0.824, 0.820, and 0.813, respectively, for the immature, ripe, and over-ripe fruit. The corresponding over-all yields were 74.7, 71.8, and 72.4%. There was no apparent relationship between the yield of processed cherries and the soluble-solids content of the 3 groups. The color of the processed product in terms of transmittance of the diluted supernatant juice, ranged from 58% for the immature to 31% for the over-ripe fruit.

TABLE 3

Effect of maturity and bruising on drained weight and yield of canned cherries

Cherries and treatment[1]	Raw cherries		Canned cherries			
	Gain in wt. during soaking	Soluble solids before soaking	Soluble solids	Color (transmittance)	Ratio of drained wt. to put in wt.	Yield
	%	%	%	%		%
Immature						
unbruised	3.7	12.1	9.7	58.0	.824	74.7
bruised	0.8	12.1	9.8	53.5	.858	72.8
Ripe						
unbruised	3.9	14.5	11.2	40.0	.820	71.8
bruised[2]	1.7	14.5	11.0	39.5	.847	74.5
Over-ripe						
unbruised	4.4	15.1	11.7	31.0	.813	72.4
bruised	0.5	15.3	11.8	27.5	.838	71.8

[1] All cherries were picked from the same tree within 2 hours and were soaked for 23 hours at 50° F. The three maturity groups were obtained by sorting the cherries on the basis of color.

[2] This sample was bruised by dropping the cherries once from a height of 3 feet. The other bruised samples were dropped twice from 3 feet.

Cherries of the 3 maturity groups responded to bruising in approximately the same manner. Bruising lessened the gain in weight during the soaking period, increased the ratio of drained weight to put-in weight, and slightly intensified the red color. The effects of bruising on yield, however, were inconclusive. The bruising treatment given the ripe cherries was less severe than that given the other samples. Data shown in Table 3 are from a single experiment.

Effects of cooling, soaking, and storing the cherries. During our studies, it was observed that the firmness of freshly picked cherries gradually increased, even though the cherries were given no experimental treatment. If cherries were compressed in a pressure tester *(8)* immediately after being picked, an appreciable quantity of juice exuded from them. If the cherries were held in air for 3 or 4 hours, however, the same pressure caused no exudation of juice. This observation suggested the following experiment, in which some cherries were pitted and processed within 2 hours after being picked, and others stored in air or in water for 15 hours before being processed.

The pitting loss (18.3% at about 75° F.) from freshly picked fruit was relatively high (Table 4). Storing the cherries in air at 65° F. (18.3° C.) for 15 hours reduced their pitting loss to 15.3%. On the other hand, soaking the cherries in water at 65° F. for 15 hours increased their pitting loss to 19.2%. Cherries bruised before soaking, and others that were bruised after soaking, had lower pitting losses than similar unbruised soaked cherries.

The pitting loss from soaked cherries cannot logically be compared with the loss from the original cherries, because the 2 samples differed in age. By comparing the behavior of soaked cherries with the behavior of similar cherries stored in air, the effect of age is at least partly cancelled out, and the increase in pitting loss attributable to soaking becomes apparent. The effect of temperature alone on pitting loss is not indicated reliably by the data of this experiment, since it is not possible to distinguish between the effects of temperature and aging. Some of the changes in cherries associated with aging have been described previously *(9, 10)*.

Storing the raw cherries in air or in water for 15 hours at 65° F. (18.3° C.) increased both the yield of the canned product and the tenderometer reading. Increases were greater with the soaked cherries than with the cherries stored in air. The soaked cherries, however, decreased in soluble-solids content, whereas there was no change in the soluble-solids content of the fruit stored in air. None of the cherries changed appreciably in color during the 15 hour treatment.

A number of observations may be made concerning cherries which were of the same post-harvest age but which were stored at different temperatures. Cherries stored in air or in water at 35° F. (1.7° C.) gave lower average yields, ratios of drained weight to put-in weight, and tenderometer readings than did similar cherries stored at 65° F. For instance, the average yields of all cherries stored at 35, 50, and 65° F. were 72.3, 73.7, and 74.7%, respectively, and the corresponding average tenderometer readings were 25.9, 26.0, and 27.5. Cherries stored (in air or in water) at 35° F. were relatively stable, and their behavior during processing approximated that of the original fruit. The greatest changes occurred in the samples stored at 65° F. Bruising the cherries, either before or after soaking, made almost no difference in the yield of processed fruit, although bruising did increase the tenderometer reading. Bruising produced no cull cherries in any of the experiments reported in this paper.

TABLE 4

Effects of soaking, temperature, storage in air, and bruising on the yield and quality of canned cherries

Treatment of raw cherries[1]	Pitting loss[2] (raw cherries)	Soluble solids	Tenderometer reading	Color (transmittance)	Ratio of drained wt. to put in wt.	Yield
	%	%	lbs./in.²	%		%
None. Processed 2 hrs. after harvest.						
About 75° F.	18.3	11.5	23.1	41.0	.881	72.1
Stored in air						
35° F.	15.0	11.4	26.8	40.0	.847	71.6
50° F.	13.9	11.1	25.5	40.0	.858	73.6
65° F.	15.3	11.5	25.7	40.0	.877	73.6
Soaked in water						
35° F.	18.5	11.2	25.0	40.0	.847	73.2
50° F.	17.8	11.2	23.5	40.0	.842	74.2
65° F.	19.2	11.2	27.3	40.5	.856	75.2
Bruised and soaked						
35° F.	17.8	11.0	25.0	40.0	.865	73.1
50° F.	16.2	11.0	31.1	39.5	.854	73.2
65° F.	17.1	11.2	29.9	40.5	.878	74.7
Soaked and bruised						
35° F.	17.8	11.2	28.7	40.5	.833	71.4
50° F.	17.7	11.1	24.0	40.5	.842	73.6
65° F.	16.9	11.0	26.9	41.0	.852	75.1

[1] The duration of treatments was 15 hours.

[2] The cherries were not pitted at a constant temperature.

DISCUSSION

Although the present study shows that bruising fresh cherries definitely affected their behavior during processing, bruising was studied only under a relatively narrow set of conditions. By contrast, bruising in commercial practice occurs under a wide range of conditions. Cherries may be bruised as they are picked, as they are dumped into lug boxes, as they are loaded and hauled to the processing plant, and as they are soaked, sorted, and conveyed throughout the plant. They may be held for irregular intervals at varying temperatures between the periods of bruising. It would not be proper, therefore, to interpret the effects of bruising in all or even in the majority of commercial cherries in terms of the present results. It is probable that bruising in commercial practice has several different effects, depending largely on the total post-harvest history of the various samples. Conditions have been described *(9)*, for instance, under which bruising may result in either an increase or decrease in the yield of final product. Similar differences in the effects of bruising on color, color defects, texture, soluble-solids content and flavor may occur also.

Effects of temperature and storage on the behavior of cherries during processing also were studied under only a relatively few experimental conditions. It might be expected, for instance, that greater differences among the samples of Table 4 would have been obtained if the experiment had been permitted to run for 30 hours instead of 15. In commercial practice, occasional lots of cherries are held for 48 hours or more between the times

of harvesting and processing. Obviously, further studies on the effects of bruising, cooling, and storing cherries are desirable.

SUMMARY

Although unbruised cherries gained weight when soaked in cool water and bruised cherries showed little if any gain, the final yields of canned fruit from the unbruised and bruised samples were similar. The gain of the unbruised cherries was lost during pitting and canning.

Bruising the raw cherries increased both the ratio of drained weight to put-in weight and the tenderometer reading of the processed fruit. The ratio was not a valid criterion of yield, as based on the weight of the original whole, fresh cherries. Under the experimental conditions employed, bruising had only a slight effect, if any, on the color and soluble-solids content of the final product.

Cherries ranging in color from orange to mahogany red and in soluble-solids content from 12.1 to 15.3% were picked from a single tree on the same day. There was no relationship between maturity (as based on color) and drained weight, and all cherries responded to bruising in approximately the same manner.

Cherries processed immediately after being picked gave both a low tenderometer reading (cherries were soft) and a low yield of canned fruit. Storing the fresh cherries in air or in water at 65° F. (18.3° C.) for 15 hours increased the tenderometer reading and the yield. Fruit stored at 35° F. (1.7° C.) for 15 hours gave tenderometer readings and yields not much different from those of the original fruit. Although fruit stored in air had the lower pitting loss, fruit soaked in water gave the higher yield of final product.

In conclusion it may be stated that although bruising affected the behavior of cherries during various processing steps, it had no effect on final yield and only a slight effect on quality.

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Amylases: Their Kinds and Properties and Factors Which Influence Their Activity[a]

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To select an enzyme suitable for use in the industrial processing of foods, a thorough knowledge of the properties of the enzymes under consideration is essential. Three types of amylases are discussed and serve to illustrate some of the factors which determine their value for specific applications.

The enzymes are organic catalysts specific in the reactions which they accelerate and which cause reactions to occur under milder conditions than would otherwise be possible. Because of these properties they are particularly suitable for use in the processing of foods, and they have been used successfully in many industrial processes (*10, 11*). In the development of any new process involving the use of enzymes or in the application of established enzymatic processes to new food products, a thorough knowledge of the properties of the enzyme is essential for their efficient use. It is the purpose of this paper to present basic information on the factors which influence the activity of enzymes and which determine the suitability of an enzyme for a specific industrial application. The amylases, or "diastases" as they are known commercially, are enzymes which hydrolyze starch, and have been selected for discussion because they serve to illustrate many of the problems involved in the use of enzymes.

Most starches are heterogeneous, consisting of at least 2 components, amylose and amylopectin, with glucose as the basic unit of both components. Amylose is an unbranched polysaccharide with all of the glucose units joined through 1-4, α glucosidic linkages; amylopectin is a branched chain polysaccharide with 1-6, α

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glucosidic linkages at the points of branching in addition to the 1-4, α glucosidic linkages. The complex nature of starch permits various pathways for its hydrolysis, and it is important to recognize that the resulting hydrolytic products will depend upon the specific amylase used.

The amylases occur in animal tissues and in higher plants and are produced by a large number of microorganisms. Much basic information concerning the amylases has been obtained with purified substrates and with crystalline or highly purified amylases. However, for industrial applications the use of pure enzyme preparations is neither desirable nor economically feasible. Many of the commercial amylase preparations contain small amounts of maltase, and other carbohydrases may also be present. The evaluation of an enzyme for a specific application must take into consideration the possible influence of these accompanying enzymes. Therefore, the data that have been selected to illustrate the problems under discussion were obtained with preparations similar to those produced commercially. Although some amylases are capable of hydrolyzing raw starch, they are relatively inefficient in this respect, and this discussion is confined to the action of amylases on gelatinized starch. Three types of amylases will be considered.

THE ALPHA AMYLASES

A group of enzymes known as alpha amylases has been most widely used to date and includes the alpha amylase of malted barley, pancreatic amylase, and the amylase of *Aspergillus oryzae*. All of these amylases cause a marked decrease in the viscosity of starch pastes and a rapid fragmentation of starch to reducing dextrins which give no color with iodine. They are capable of rupturing not only the 1-4, α glucosidic linkages far from the end groups in the starch molecule but also those between the points of branching. They do not hydrolyze the 1-6, α glucosidic linkages. They are called alpha amylases because the products formed by several of these enzymes exhibit falling or alpha-mutarotation. Because these enzymes have many properties in common, the fact that they also differ in many respects is often overlooked.

In Table 1 are data showing the rate of hydrolysis of starch by 3 microbial alpha amylases as determined by the increase in

TABLE 1

Rate of hydrolysis of starch by three alpha amylases [b]

Time hours	% Theoretical maltose		
	B. subtilis [c]	*B. polymyxa* [d]	*A. oryzae* [e]
½	16.4	17.6	20.7
1	25.7	36.6	36.5
2	32.0	56.0	54.0
4	37.4	63.0	67.1
24	46.0	69.0	84.6
48	50.8	72.6	94.7

[b] 2% soluble starch, 40° C., 0.05 M acetate buffer.
[c] Hydrolysis at pH 5.7.
[d] Hydrolysis at pH 6.8.
[e] Hydrolysis at pH 5.0.

the reducing value of the substrate. Although the extent of the hydrolysis occurring in 30 minutes is similar for all 3 preparations, marked differences are observed in the later stages of the hydrolysis. The extensive hydrolysis produced by the amylase of *Aspergillus oryzae* is not due entirely to accompanying carbohydrases because similar results have been obtained with highly purified preparations of this enzyme *(12)*. During the early stages of hydrolysis by alpha amylases the reducing value of the substrate is due chiefly to the presence of reducing dextrins although it is customary to calculate the reducing value as maltose. Maltose and small amounts of glucose do appear eventually in the hydrolysates of these enzymes but the point in the reaction at which they first appear and the relative amounts of these simple sugars vary with the type of alpha amylase preparation used. For further information, anyone interested may wish to refer to some of the recent review articles on this subject *(1, 5, 7)*.

Further evidence of differences among the alpha amylases is seen in Table 2. The activity of alpha amylases may be determined not only by following the reducing value of the hydrolysate (saccharogenic activity) but also by measuring the amount of enzyme necessary to hydrolyze starch to reducing dextrins which give no blue color with iodine (dextrinogenic activity). For the comparisons in Table 2 the saccharogenic activity was determined by measuring the amount of enzyme necessary to liberate in 30 minutes at 40° C. reducing products equal to 20% theoretical maltose. The dextrinogenic activity was obtained by determining the amount of enzyme necessary under similar conditions to hydrolyze soluble starch to products which give a clear red color with iodine. The ratios of these two types of measurements for the four alpha amylases are seen to differ considerably. These results indicate that alpha amylases differ in the way that they break down starch, even during the early stages of hydrolysis. The data in Tables 1 and 2 also serve to illustrate the importance of evaluating enzyme preparations from different sources by methods which relate as closely as possible to the desired end point.

TABLE 2

Comparison of the dextrinogenic to the saccharogenic activity of various alpha amylases

Amylase	D/S
Pancreatic [f]	2.6
A. oryzae [g]	7.5
B. polymyxa [h]	2.2
B. subtilis [i]	6.5

[f] Measurements in presence of 0.02 M NaCl and at pH 7.0.
[g] pH 5.0.
[h] pH 6.8.
[i] pH 5.7.

The alpha amylases have been particularly useful in processes where the presence of gelatinized starch impedes filtration and efficient extraction. They have also proved useful in the clarification of fruit juices which are turbid due to the presence of starch. Since these amylases all cause a rapid disintegration of starch, the enzyme of choice will depend upon the particular substrate to be used and upon other environmental conditions which will be considered later. For fermentation processes it is essential to use an enzyme which causes an extensive hydrolysis of starch to simple sugars such as is observed with the amylase of *Aspergillus oryzae*. For rapid liquefaction with minimum production of sugar, an enzyme such as the amylase of *Bacillus subtilis* should be selected.

BETA AMYLASES

A second type of amylase, beta amylase, is found in several of the cereal grains, in soybeans and in sweet potatoes. These enzymes hydrolyze starch by liberating maltose from the nonreducing ends of the glucosidic chains and are unable to attack 1-6, α glucosidic linkages. They are called beta amylases because they liberate maltose as beta maltose; the term saccharogenic amylase is frequently applied to them. They hydrolyze amylose completely to maltose but amylopectin only partially by the liberation of maltose from the ends of the branches until the action of the enzymes is stopped by the 1-6, α glucosidic linkages. They exert little influence on the viscosity of starch paste and hydrolyze most starches to approximately 60% maltose, at which point the reaction mixtures still give a blue violet or violet color with iodine. Alpha amylases supplement the action of beta amylases by liberating additional end groups which are then subject to attack by beta amylase. Alone, beta amylases have proved of little industrial value to date but a combination of alpha and beta amylases is widely used in the brewing and fermentation industries and in the baking industry.

GLUCOSE-LIBERATING AMYLASES

A third type of amylase found in several fungal preparations has recently received considerable attention. These amylases resemble the beta amylases in many respects but liberate glucose instead of maltose, and are capable of hydrolyzing starch almost completely. Various names have been suggested for these enzymes such as amyloglucosidase *(3)*, gluc amylase *(8)*, and gamma amylase *(4, 11)*. These enzymes hydrolyze maltose as well as starch, and Corman and Langlykke *(2)* consider them maltases. In Table 3 are data showing the rate of hydrolysis of

TABLE 3

Hydrolysis of starch by amylase [1] of Rhizopus delemar

Time hours	% Theoretical glucose
1	11.3
2	21.1
3	31.1
4	38.2
5	48.4
6	54.0
24	83.0
48	90.0

[1] 40° C., pH 4.5.

starch by the amylase produced by the fungus, *Rhizopus delemar*. The extensive hydrolysis noted, 90% in 48 hours, indicates that this enzyme is not hindered by the 1-6, α glucosidic linkage. Phillips and Caldwell *(9)* have obtained indirect evidence suggesting that this enzyme by-passes this linkage in some way without actually hydrolyzing it. Because of their high sugar formation these enzymes are suitable for use in fermentation processes and were used for saccharification in a process of alcoholic fermentation known as the Amylo process many years before their mode of action was understood.

FACTORS INFLUENCING ACTIVITY OF THE AMYLASES

In selecting an enzyme suitable for a specific application, it is necessary to consider not only the desired end point but also the need for working under defined environmental conditions. The enzymes are particularly sensitive to the pH of the substrate on which they act. Table 4 gives the optimum pH obtained under comparable conditions with several amylases. These optima hold only for the conditions specified, and are shown merely to illustrate the differences in the optimum pH of amylases from different sources. Many factors influence the optimum pH of an enzyme, such as temperature, the presence of stabilizing agents, the concentration of substrate, and the length of time of the hydrolysis.

Temperature is another factor which must be carefully controlled if enzymes are to be used successfully. Enzymes are heat labile, but at the same time the rate of the reaction catalyzed by the enzyme is increased as the temperature is elevated. Some alpha amylases are much more heat labile than others. The activity of the

TABLE 4

Optimum pH for the activity of various amylases

Kind	Optimum pH	Condition	
		Buffer	General
Pancreatin	7.0-7.2	Phosphate	
A. oryzae	5.0-5.5	Acetate	
R. delemar	4.0-4.5	Acetate	30 minutes hydrolysis
Malted barley alpha	4.5-4.7	Acetate	40° C.
Malted barley beta	4.5-4.7	Acetate	2% soluble starch
B. subtilis	5.3-5.7	Acetate	
B. polymyxa	6.7-7.0	Phosphate	

amylase of *Aspergillus oryzae* decreases rapidly at temperatures above 60° C. whereas the amylase of *Bacillus subtilis* can be used at temperatures of 75°-85° C. and in concentrated starch solutions maintains some activity for a short period of time when heated at 100° C. A heat-labile amylase may be very useful for certain types of processing because of the ease with which its activity can be stopped if a specific end point is desired. On the other hand, the value of an amylase that can act at the high temperatures which are necessary for the gelatinization of concentrated starch slurries is readily appreciated. Here again it must be emphasized that the influence of temperature on the activity of an enzyme is dependent on many factors and must be determined for the particular system in which the enzyme is to be used.

Most enzymes show their greatest stability in neutral solutions and the optimum pH for their activity approaches neutrality as the temperature is elevated. There are many other factors that influence the stability of an enzyme. High concentrations of substrate often favor their stability. Certain inorganic ions such as calcium are particularly effective in increasing the stability of alpha amylases. The presence of protein or of amino acids may also prove beneficial.

The last factor to be considered is the presence of substances inhibiting the enzyme. Calcium ions increase the heat lability of beta amylases. The presence of a small amount of a heavy metal such as copper may reduce or completely inhibit the activity of an enzyme. Many of the detergents will retard or inactivate the enzymes. Specific inhibitors for the amylases have been reported in certain cereals *(6)*. Such inhibitors can be troublesome but if the problems are recognized there are often many methods for their solution. For example, pancreatic amylase is sensitive to traces of copper but the presence of a small amount of phosphate will usually overcome the difficulty by removing the copper ion from solution. There are detergents that can be used successfully without interfering with the action of an enzyme. Some of the amylase inhibitors are effective on amylases from one source but not from another *(6)*. Their presence in a food product may necessitate the elimination of certain enzymes for processing a specific food but other enzymes may be available which are equally satisfactory and which are not susceptible to this inhibition.

Table 5 illustrates the importance of considering the many factors involved when evaluating an enzyme for possible application. At pH 5 and at 40° C. sucrose is seen to reduce appreciably the activity of the amylase of *Aspergillus oryzae*. At 57° however, the activity in the presence of 30% sucrose is practically equal to that observed for the control at 40°. Although activity equal to the control was not observed in the presence of 40% sucrose under any of the conditions studied, it is possible to obtain 70% of the control activity if the temperature is elevated to 63°. These data illustrate an additional point. At 40° C. the optimum pH of this amylase is 5.0. At 63°, however, it is seen that the activity in the absence of sucrose is much greater at pH 6 than at pH 5. This is an example of an enzyme that is more stable in neutral than in acid solutions, and therefore as the temperature is elevated the optimum pH approaches neutrality. On the other hand, in the presence of sucrose which is apparently stabilizing the enzyme, the activity at pH 5 is as good or better than that at pH 6 for all of the concentrations of sucrose studied, regardless of the temperature.

TABLE 5

Influence of sucrose on activity of the amylase of Aspergillus oryzae[a]

Conditions	pH 5			pH 6	
% Sucrose	40° C.	57° C.	63° C.	57° C.	63° C.
0	100[b]	84	35	81	76
20	62	99	69	74	73
30	44	91	82	85	68
40	24	62	70	45	48

[a] 2% soluble starch.
[b] Activity in absence of sucrose at 40° and pH 5.0 = 100%.

SUMMARY

In summary, it is important to realize in selecting an enzyme for a specific application that enzymes hydrolyzing the same substrate may do so in a variety of ways, as was illustrated by the action of different amylases. The following factors must be considered: a, the specific end point desired; b, the optimum pH of the activity of the enzyme in relation to the natural pH of the food to be processed; c, the temperature at which it is desirable to process the food; and d, the presence in the food of stabilizing or inhibiting substances. With each new application it is necessary to spend some time in selecting the most suitable enzyme system to be used, and in developing the conditions necessary for its most efficient action. Once a process is developed, enzymes have proved to be useful tools and permit much more satisfactory control than is possible by the usual methods of hydrolysis.

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A Comparison of Cereal, Fungal, and Bacterial Alpha-Amylases as Supplements for Breadmaking[a,b]

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Cereal, fungal and bacterial amylase preparations have been compared as supplements for breadmaking. The firmness of the bread crumb as well as the quantity and characteristics of the dextrin extracted from the bread stored for 90 hours appears to be largely associated with the thermostability of the enzymes employed. Bacterial alpha-amylase is most thermostable, cereal alpha-amylase possessed intermediate thermostability and fungal alpha-amylase was the least thermostable. Bacterial amylase tended to be protected against thermal inactivation by the presence of starch. The other enzymes did not exhibit this characteristic. Increasing concentration of each enzyme type decreased the rate of firming of bread crumb and the dextrins tended to become more complex. All amylases caused the amount of extractable dextrin in bread to increase but bacterial amylase was much more effective than fungal or cereal amylase. The dextrins tended to be less complex as bread aged.

The historical and contemporary enzyme supplement for baked goods is malted wheat or barley flour. Considerable interest has developed recently in the use of enzyme concentrates produced by culturing certain fungi and those enzymes produced by bacteria. Standards of identity for bread proposed by the Federal Security Agency *(18)* provide that enzyme preparations from the fungus, *Aspergillus oryzae*, may now be used in commercial bread production. They may not as yet be added to flour at the mill. Bacterial preparations are not permitted for use as enzyme supplements in baking.

Enzyme supplements are useful in baking primarily because of the two enzyme systems, alpha-amylase and proteinase, which they contain. These enzymes function during two phases of the baking process. During fermentation the amylase hydrolyzes a small amount of available starch (approximately 3% while proteinase reacts with the gluten proteins to modify the physical dough characteristics. During baking the proteinase is inactivated at relatively low temperatures (50°-60° C.) *(17)*. The amylase, depending upon its source, modifies the gel properties of the starch that is made available to the amylases through gelatinization during the baking in the oven. The extent of starch modification during the baking process differs with cereal, fungal, and bacterial alpha-amylase and also with enzyme concentration.

Several workers, including Green *(6)*, Harrel, Lincoln, and Gunderson *(7)*, Johnson and Miller *(12)*,

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Kneen and Sandstedt *(14)*, and Miller and Johnson *(16)* have suggested that fungal enzymes are suitable for use in breadmaking. Advantages include the ease of regulating amylolytic as well as proteolytic activity and an increase in softness of crumb with the elimination of sticky or gummy crumb. Fungal enzyme supplements also permit development of a finer grain and texture, better color, and uniformity. The ability to regulate the quantities of both alpha-amylase and proteinase is perhaps the greatest single advantage in favor of the use of fungal preparations since the concomitant proteinase is mainly responsible for the dough consistency and its ability to machine properly. These preparations are being marketed today in forms ranging from the concentrate to blends of the enzymes with yeast food produced in tablet form. Many large commercial bakeries regularly use these enzyme preparations with good success.

Kozmin *(15)*, working with sprout-damaged wheat, concluded that stickiness in bread crumb was due to excessive enzymic splitting of the starch during the baking process. "The crumb appears wet and sticky not because there is too much water but because this water is not held together sufficiently well by the gel of the starch and forms with the dextrins and sugars a stickiness."

Bacterial amylases have shown little promise as diastatic supplements *(4, 12)* because they produce a sticky and gummy crumb. This is generally recognized as being due to the high thermostability of bacterial amylases which have been observed to survive a short boiling in the presence of starch *(20)*. Hopkins and Kulka *(9)* suggested that a difference in the end products of starch hydrolysis by bacterial and malt alpha-amylase might be expected when based on the affinity of the respective enzymes for the substrate in the early stages of the reaction. Bernfeld and Studer-Pécha *(2)* confirm this hypothesis. The quantity of dextrins produced by different amylases hence might be a function of the length of time that the amylase remains active during baking and their affinities for the high molecular weight substrate.

One of the most challenging problems confronting the baking industry today is one of developing means of controlling the rate of bread staling. While bread staling is probably a complex phenomenon, physical changes in the starch structure are of utmost importance. It is possible to change starch structure by enzymatic modification. Jackel et al. *(10)* have proposed the use of bacterial amylase as a means of retarding the staling of bread.

In spite of the interest shown in the application of various types of alpha-amylase to breadmaking, there have been few attempts *(4, 13)* to compare the action of the different enzymes under comparable bake shop conditions. The different properties of these enzymes, make them worthwhile subjects of comparative study.

The object of the present work has been to determine the change in bread firmness with time and to relate this change to the quantity of dextrin and the average chain length of dextrin extracted from bread baked with various levels of cereal, fungal, and bacterial amylase.

MATERIALS AND METHODS

An unmalted commercial hard red winter, straight grade flour having a protein content of 11.2% (14% moisture basis) was used for all experiments.

The enzyme supplements included a commercial malted wheat flour and two commercial enzyme concentrates, one of which was of fungal (*Aspergillus oryzae*) origin[4] and the other of bacterial origin.[5] The alpha-amylase activity for these preparations was 74, 5000, and 384 SKB units *(19)* per g., respectively.

The sponge dough baking procedure reported by Johnson and Miller *(11)* was employed. The bread was allowed to cool for 1 hour and sealed in plastic bags until needed.

Crumb firmness was determined with a Bloom gelometer after storage of the bread for periods of 18, 42, 66, 90, and 114 hours. The experimental value for firmness was expressed in grams of lead shot required to depress a 1-inch diameter plunger 4 mm. into a slice of bread. Two determinations were recorded for each of 3 slices from duplicate loaves.

The quantity of dextrin in the baked bread was determined in samples prepared by mixing 100 g. of bread crumb with 500 ml. of water in a Waring blendor for 10 minutes. Bread was extracted after being stored for 1, 18, 42, 66, and 90 hours. The homogenate was centrifuged for 25 minutes at 1500 r.p.m. in an International Model 2 centrifuge and autoclaved for 20 minutes to destroy any residual enzyme activity. The centrifugate was fermented for 18 hours with bakers yeast and recentrifuged. Brown and Morris *(3)* showed that dextrins and maltodextrins were not fermented by the yeast *Saccharomyces cerevisiae*. Bacterial growth was prevented by autoclaving before fermentation. Quantities of 95% ethyl alcohol ranging from 50 to 175 ml. were added to duplicate 50-ml. aliquots of this dextrin solution and the mixture allowed to stand in the refrigerator overnight. One of the precipitated 50-ml. aliquots of dextrin was filtered through a Gooch crucible, dried for 2 hours at 130° C. and weighed. The supernatant liquid was decanted from the duplicate flask and the precipitate washed with water into a 70-ml. portion of water. The mixture was then heated to boiling, diluted to 100 ml. and filtered. Aliquots of this material were analyzed for reducing groups *(1)* both before and after hydrolysis to maltose by autoclaving for 30 minutes in the presence of 0.5 *N*. sulfuric acid. Preliminary experiments indicated that these conditions were adequate to release the maximum amount of maltose. The data also permitted a calculation of the average chain length of the extracted dextrin. It should be recognized, however, that the figure obtained for average chain length is merely an expression of convenience. A given dextrin conceivably can consist of larger molecules than is indicated by the increase of reducing groups following hydrolysis.

Alpha-amylase dextrinization activity was determined by the procedure described by Sandstedt, Kneen, and Blish *(19)*. Various concentrations of the enzymes were diluted to 470 ml. with sodium acetate-acetic acid buffer (pH 5.55) containing 0.2% calcium chloride and heated in the Amylograph. Experiments were performed both with and without the addition of flour. The stability of the various enzymes was determined by analyzing the centrifugate obtained from aliquots taken from the Amylograph at temperature intervals ranging from 30° to 95° C.

RESULTS AND DISCUSSION

The effects of supplementation by various levels of malted wheat flour, fungal, and bacterial alpha-amylase on crumb firmness measured over a 5-day period are shown in Figures 1 and 2. Bread baked with a normal amount of malted wheat flour supplement and with equivalent units[6] of fungal and bacterial amylase produced the results shown in Figure 1. The bacterial amylase was much more effective than either of the

[4] A blend of Rhozyme-S and Diastase-33, Rohm and Haas Co., Philadelphia, Pa.

[5] Rhozyme-DX, Rohm and Haas Co., Philadelphia, Pa.

[6] It is recognized that equivalent unitage is based on a determination at 30° C. and does not account for variations in thermostability of the enzyme or the activity at high temperatures.

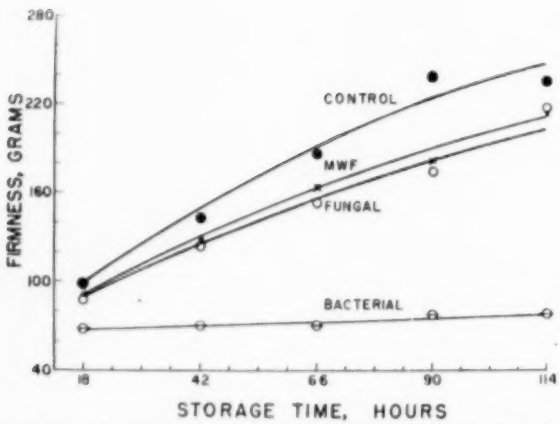

Figure 1. A comparison of the effects of equivalent levels malted wheat flour, fungal, and bacterial alpha-amylase on bread firmness.

other two sources of amylase in decreasing the rate of bread staling as measured by firmness of the crumb. The bread baked with the bacterial preparation, however, was very sticky and gummy. All types of amylase reduced the rate of firming as compared with the control containing no added supplement.

The data in Figure 2 show that increasing concentrations of all types of amylase produced a steady decrease in the rate of firming. The firming of the bread baked with 24 bacterial alpha-amylase units per loaf increased as the bread aged up to 3 days. The firmness of the bread remained constant from 3 to 5 days. The use of 210 alpha-amylase units from the bacterial source (Figure 1) resulted in no change in bread firmness in the period of 1 to 5 days. Additional results were obtained which indicated that it is also possible to add sufficient fungal amylase (4800 units per loaf) to prevent any change in the bread firmness over a two-day storage period.

Thermostability of enzyme supplements. Most enzyme systems are known to be more stable in the presence of their substrates. In order to simulate the actual baking conditions more closely, appropriate quantities of the different enzymes were heated in the amylograph in the presence and absence of flour. Aliquots were taken at definite temperatures, cooled and analyzed. The results of these analyses are shown in Tables 1 and 2.

The substantially greater stability of the bacterial amylase heated in the presence of flour (Table 1) could not be demonstrated for malted wheat flour and a fungal

TABLE 1

Thermostability of bacterial amylase

Temperature	Retention at enzyme concentrations (Alpha Amylase Units per 470 ml. liquid) of			
	187 [a]	93 [a]	187 [a]	374 [a]
°C.	%	%	%	%
30	100	100	100	100
75	100	100	100	100
80	92	100	100	100
85	58	92	100	100
90	22	49	80	100
95	8	27	26	62

[a] No flour present.
[b] In the presence of 50 g. of flour.

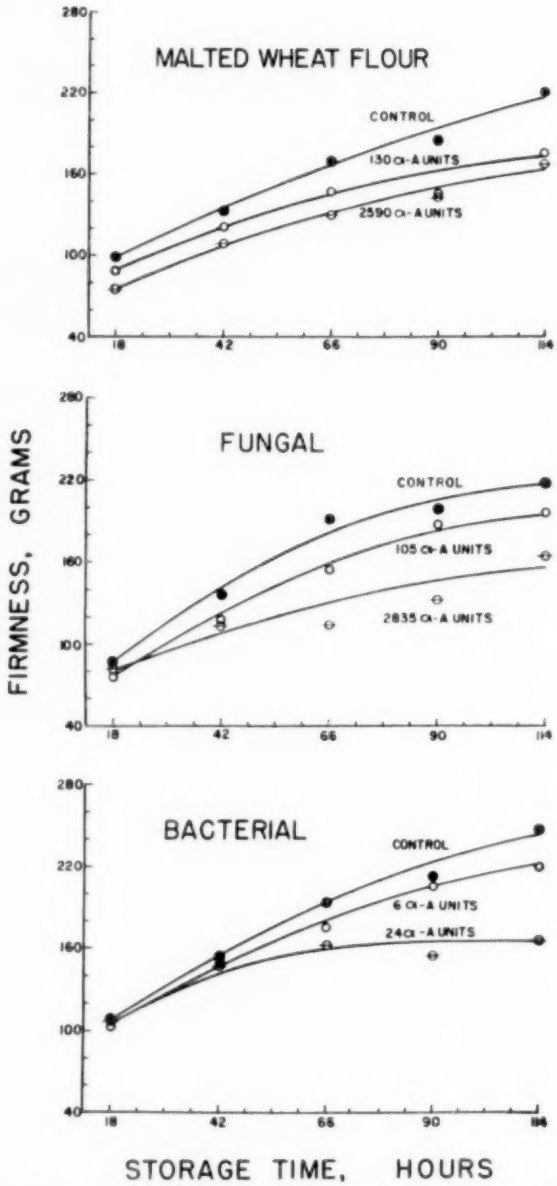

Figure 2. A comparison of the effects of different levels of malted wheat flour, fungal, and bacterial alpha-amylase on bread firmness.

amylase treated under similar conditions. Other bacterial preparations including a concentrate containing no inorganic salts showed similar stability.

The data shown in Table 2 demonstrate the relative thermostabilities of the three sources of amylase when heated in the absence or presence of flour. These data offer a reasonable explanation for the differences in bread firmness shown graphically in Figure 1. The bacterial amylase is definitely the most thermostable while the fungal amylase is the most thermolabile of the three types of amylases studied.

Quantity of extractable dextrin. As an additional means of studying the differences observed in Figure 1,

TABLE 2

Comparison of the thermostability of fungal, malted wheat flour (MWF), and bacterial alpha-amylase at comparable levels of activity

Temperature	No flour			50 g. flour per 470 ml. liquid		
	Enzyme retention			Enzyme retention		
	Fungal	MWF	Bacterial	Fungal	MWF	Bacterial
°C.	%	%	%	%	%	%
30	100	100	100	100	100	100
60	100	100	100	97	100	100
65	100	100	100	83	100	100
70	52	100	100	52	92	100
75	3	58	100	11	69	100
80	1	25	92	3	29	100
85	—	1	58	—	2	100
90	—	—	22	—	—	80
95	—	—	8	—	—	26

the quantities of dextrins extractable from the baked bread by water were determined. The data in Table 3 indicate that the fungal and malted wheat flour amylases caused only a slight increase in the quantity of extractable dextrins while the bacterial amylase increased the quantity by more than 5-fold. The quantity of extractable dextrin tends to decrease as the bread ages, probably because of a tendency for the starch and dextrins to crystallize with age. This is demonstrated further by the data in Table 4 which indicate the increase in soluble dextrins released by increasing concentrations of alpha-amylase.

The quantity of fermentable sugar increased only slightly with increasing concentrations of the various enzymes (Table 5). The bacterial preparation and malted wheat flour produced the largest quantities of sugar. There was no significant change in the amount of sugar present in the extracts made from bread stored for 1 to 90 hours after baking.

Release of reducing groups. There were no significant differences in the average chain lengths of the dextrins produced by the fungal enzyme from those released by the natural flour enzymes (Table 6). The average chain lengths of the dextrins released by malted wheat flour tended to be somewhat longer, while the dextrins released by the bacterial preparation were considerably larger than those released by the other enzymes from bread extracted after being baked for 1 hour. This may be due to the ease of rupturing certain parts of the starch molecule *(8)* as compared with the conversion of maltodextrin to maltose. There appeared to be a general trend toward a less complex extractable dextrin as the bread aged. This may be assumed to be due to an insolubilization by crystallization of the more complex dextrin fragments as the bread ages.

In addition to the trend toward shorter and less complex dextrins as bread ages, there is also the trend toward increasing complexity as the enzyme concentration is increased (Table 7). These trends are particularly marked for bacterial amylase.

TABLE 3

Quantity of dextrin produced by equivalent quantities of various enzymes[a]

Enzyme[b]	Quantity of dextrin[c] extracted after baking at intervals of				
	1 hour	18 hours	42 hours	66 hours	90 hours
	mg.	*mg.*	*mg.*	*mg.*	*mg.*
Control	242	203	196	183	151
Fungal	319	236	234	217	149
Malted wheat flour	424	297	280	258	175
Bacterial	1868	1385	1314	1284	1146

[a] 40 SKB units per 100 g. of flour.
[b] Mg. per 10 g. of bread crumb.

TABLE 4

Quantity of dextrin produced by different levels of three sources of alpha-amylase

Enzyme	Enzyme concentration	Quantity of dextrin extracted after baking at intervals of[1]				
		1 hour	18 hours	42 hours	66 hours	90 hours
	SKB units	*mg.*	*mg.*	*mg.*	*mg.*	*mg.*
Fungal	Control	226	192	182	155	159
Fungal	105	181	187	164	146	151
Fungal	315	298	227	217	177	209
Fungal	945	588	357	392	273	278
Fungal	2835	1187	1003	1210	652	808
MWF	Control	211	181	171	168	153
MWF	131	309	271	207	200	
MWF	796	667	441	273	378	296
MWF	1295	663				
MWF	2590	948	636	501	501	466
Bacterial	Control	206	189	198	174	180
Bacterial	3	332	311	363	288	338
Bacterial	6	541	492	569	475	609
Bacterial	12	1001	997	985	767	1089
Bacterial	24	1314	1345	1359	1151	1450

[1] Each value is the average of 3 results obtained by precipitating 50 ml. of extract with 75, 100, and 175 ml. of ethyl alcohol.

TABLE 5

Gas production of water extracts of bread baked with various levels of cereal, fungal, and bacterial enzymes

Enzyme	Enzyme concentration	Average gassing power
	SKB units	*mm.*
Control	0	66
Fungal	40	68
MWF	40	80
Bacterial	40	115
Control	0	63
MWF	131	70
MWF	796	88
MWF	1295	91
MWF	2590	99
Control	0	55
Fungal	105	71
Fungal	315	73
Fungal	945	71
Fungal	2835	72
Control	0	64
Bacterial	3	67
Bacterial	6	70
Bacterial	12	71
Bacterial	24	73

TABLE 6

Average chain lengths of dextrins produced by equivalent quantities of alpha-amylase from various sources

Enzyme[a]	Average chain lengths of dextrins extracted after baking at intervals of				
	1 hour	18 hours	42 hours	66 hours	90 hours
Control	30	18	24	20	20
Fungal	26	26	31	21	23
Malted wheat flour	43	27	26	28	22
Bacterial	50	29	20	21	19

[a] 40 SKB units per 100 g. of flour.

SUMMARY AND CONCLUSIONS

Three types of enzyme supplements added to Hard Red Winter Wheat flour were compared. These were a commercial malted wheat flour and two commercial

TABLE 7

Average chain lengths of dextrins produced by increasing quantities of alpha-amylase from various sources

Enzyme	Enzyme concentration	Average chain lengths of dextrins extracted after baking at intervals of				
		1 hour	18 hours	42 hours	66 hours	90 hours
	SKB units					
Fungal	Control	15	15	9	7	10
Fungal	105	12	12	9	6	6
Fungal	315	15	13	8	6	9
Fungal	945	20	17	15	8	11
Fungal	2835	32	22	24	16	23
Malted wheat flour	Control	12	7	8	10	6
Malted wheat flour	131	17	10	7	11	7
Malted wheat flour	796	25	17	10	12	10
Malted wheat flour	1295	20	16			
Malted wheat flour	2590	22	22	15	17	10
Bacterial	Control	26	23	20	23	17
Bacterial	3	35	30	26	27	25
Bacterial	6	40	37	37	34	32
Bacterial	12	53	48	38	38	33
Bacterial	24	65	50	40	36	37

enzyme concentrates, one of fungal (*Aspergillus oryzae*) origin and the other of bacterial origin. All three types of enzymes decreased the rate at which bread becomes firm with age. The bacterial enzyme was much more effective; however, this enzyme caused the bread crumb to be sticky and gummy when sufficient quantities were used to maintain a soft crumb for several days. Increasing quantities of all enzymes produced a steady decrease in the rate of firming. Large amounts of fungal amylase maintained the same degree of softness as did bacterial amylase. Approximately 100 times more fungal amylase units were required than bacterial units.

The fungal amylase was the least and the bacterial amylase the most thermostable of the three preparations. The bacterial preparation was even more thermostable in the presence of a flour substrate and the thermostability increased as the amount of enzyme per given quantity of flour was increased. The same thermostability resulted with the presence or absence of stabilizing inorganic salts. The thermostability of fungal and malted wheat flour alpha-amylase did not appear to be affected by the substrate.

The quantity of extractable dextrins produced was much greater for bacterial amylase, than for fungal or malted wheat flour amylases. The quantity of dextrins increased steadily with increasing levels of each enzyme. In all cases, the extractable dextrins decreased markedly with the age of the bread. The bacterial and malted wheat flour preparations produced somewhat larger quantities of sugar than the fungal preparation. The bacterial enzyme produced the largest amount per unit of alpha-amylase.

The dextrins produced with bacterial amylase were more complex than those obtained by using either malted wheat flour or fungal amylase. The general trend toward a less complex extractable dextrin as bread aged was attributed to a retrogradation of the dextrin fragments. The dextrin from the bread stored for 90 hours which showed no significant change in firming with time was of essentially the same complexity as that from bread which became markedly more firm with time. The firming of bread with time appears largely as a function of the extent of starch conversion and only partly a function of the nature of the conversion products. Starch conversion is related to thermostability and enzyme concentration.

The comparative data for the three alpha-amylase sources confirm the unusual nature of bacterial amylase (*5, 9*). The difference in behavior can be explained largely by the higher degree of thermostability of the bacterial amylase. The bacterial enzyme concentrate, when freed of any stabilizing inorganic salts, showed the same degree of thermostability. The data indicate that the bacterial amylase employed holds little promise as an enzyme supplement in baking because of its tendency to produce a sticky and gummy crumb. It is possible, however, that certain other bacterial amylases, when properly controlled as to type and quantity, might have value as enzyme supplements for baking.

Acknowledgment

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Comparison of Methods for Measuring the Maturity Factor in Frozen Peas[a]

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Comparisons were made of the "maturities" of 68 samples of frozen peas as measured by various physical and chemical tests and as rated by panels of trained human judges. The samples represented four varieties, three seasons, and several harvest dates and size classes. Careful handling of the peas from harvesting to freezing minimized off-flavor development and other deteriorative changes. The 20 to 24 samples obtained each season were scored organoleptically for skin and cotyledon textures by means of a novel experimental design which gave results of good precision. Alcohol-insoluble solids correlated best with subjective scores, while total solids, density, and the tenderometer value of the cooked peas were somewhat inferior in this respect. The two flotation tests investigated reflected the limited reliability of density as a measure of maturity.

The texture of frozen peas is dependent in large measure on the stage of development of the raw peas used. The term "maturity" has been employed to define this property. In this paper it refers to the texture of thawed and cooked peas as determined by a trained taste panel.

It is obvious that an accurate measure of the maturity factor of frozen peas would be desirable for both research and grading purposes. Yet the use of trained panels is not only costly and time-consuming but also becomes extremely difficult where simultaneous tasting of numerous samples is attempted. The possibility of developing objective methods which correlate well with taste panel results has therefore attracted the attention of several investigators; their work has recently been reviewed in detail *(6)*. The aim of the present study was to make a comprehensive evaluation of various objective tests to determine those which correlate best with taste panel results.

Measurements were carried out on lots representing 4 harvests and 4 varieties grown in Utah in 3 successive seasons. Data on the raw material as harvested will be published separately.[c] The material used for this study was quickly and carefully handled in harvesting, shipping, and processing. The peas (in pod) were kept cool, and the time between shelling and blanching was held to a minimum to avoid changes in flavor *(3, 9, 15)* or texture *(1, 9, 10)*.

MATERIALS AND METHODS

Two varieties of peas were grown each of 3 seasons at Farmington, Utah. The variety Thomas Laxton was grown all 3 seasons, with Rondo in 1949, Oracle in 1950, and Dark-Seeded Perfection in 1951. Each variety was harvested at 4 maturity levels by removal of pods from mowed vines. The pods were cooled in ice water and shipped by air to this Laboratory. At no time were the chilled pods held longer than 24 hours between harvesting and shelling. Complete temperature records were kept. The highest temperature in any 50-lb. carton at any time was 62° F. (16.5° C.); the average temperature was close to 50° F. (10° C.). Studies from this Laboratory indicate that there could have been only small changes, if any, in the texture of the processed peas from pods treated as described *(3)*. The pods were shelled in a mechanical huller and cleaned and sized in a vibrating screen cleaner. Sizes 2, 3 plus 4, 5 plus 6, and 7 were separated. Tenderometer values were obtained at once. All peas were promptly steam-blanched for 90 seconds at 212° F. (100° C.), fog-cooled, and frozen and stored at —10° F. (—23.3° C.). The time interval between shelling and blanching was less than 1 hour.

The frozen lots, representing 2 varieties and several maturity levels and size classes each season, were each well mixed before sampling for analyses. Frozen peas were used directly for tests unless thawing was indicated.

Size class 2 was lacking in the later harvests, just as size class 7 was lacking in the earlier harvests. The variety Dark-Seeded Perfection grown in 1951 did not produce enough peas of size group 7 even in its last harvest. Therefore, 24 sample lots were obtained in 1949, 24 in 1950, and only 20 sample lots were available in 1951. In some sample lots, the amounts of material in the extreme size groups were insufficient for all tests, as the omissions in the table indicate.

Weight per pea. Weight per pea was determined by dividing the weight of approximately 100 grams of frozen peas by the number. The values given are an average of 2 samples.

Tenderometer values of raw peas. The tenderometer determinations of sized lots of raw peas were made by the usual method *(10)* with 5 to 10 replications of each lot. The readings were corrected to 23° C. (73.4° F.) by using a correction of 0.5 tenderometer units per 1° C., which had been previously determined on several varieties and sizes of peas. The tenderometer values decrease with increasing temperature.

Organoleptic appraisal. The frozen samples were added to half their weight of boiling distilled water and cooked for 5 minutes after the resumption of boiling. They were drained and served to the tasters without seasoning or other additives. The judges were selected after a period of training on the same material as was used later in this study. There were no absences.

The experimental design for each organoleptic study, i.e., for the tasting of all the samples for one season, was arranged as follows: all of the 20 or 24 pea samples were arranged in the order of increasing tenderometer values for raw peas without regard to variety, and were numbered from 1 to 20 or 24 serially. For each tasting period, one of 6 combinations, each containing 10 or 12 of these numbered samples, was used. The 6 combinations were derived according to the serial numbers:

A 1, 3, 5, 7, 9, 11, 13, 15, 17, 19, (21, 23).
B 2, 4, 6, 8, 10, 12, 14, 16, 18, 20, (22, 24).
C 1, 2, 5, 6, 9, 10, 13, 14, 17, 18, (21, 22).
D 3, 4, 7, 8, 11, 12, 15, 16, 19, 20, (23, 24).
E 1, 4, 5, 8, 9, 12, 13, 16, 17, 20, (21, 24).
F 2, 3, 6, 7, 10, 11, 14, 15, 18, 19, (22, 23).

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 11, 1952.

[b] Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.

[c] This work was carried out by the Utah State Agricultural Experiment Station under contract with the U. S. Department of Agriculture as authorized by the Research and Marketing Act of 1946. The contract was supervised by the Western Regional Research Laboratory, Bureau of Agricultural and Industrial Chemistry.

Thus, at any session, half the total samples for that season were tasted, representing nearly the full range of maturities. The samples for a session were arranged in random order and assigned code numbers before being presented to the judges.

The cotyledon and skin textures of the samples were scored by 12, 13, and 16 trained, selected judges in 1949, 1950, and 1951, respectively. Score 7 indicated very immature cotyledons and very tender skins; score 1 was given to very overmature cotyledons ("grainy" or "starchy") and very tough skins. The judges were asked to try to use the entire scale of scores at each tasting session.

When the 6 combinations were used, 3 replications of each sample resulted. In 1949 each combination was scored twice, giving 6 replications of each sample, but since the experimental error was found to be small, only 3 replications were used in 1950 and 1951.

The results of the judgments were treated statistically by the analysis of variance *(14)*. The least significant difference (L.D.) was calculated for the 5 and 1% levels of significance *(13)*. L.D. P = 0.05 in the table, for example, represents the smallest difference between any two scores which may be regarded as significant at the 5% level of probability.

Brine flotation test. The percent of thawed peas *(17)*, without skins (cotyledons) that sink in 10 seconds in 13, 15 or 16% sodium chloride solutions was determined as described in the U. S. Standards for Grades of Frozen Peas *(16)*. At least 200 peas were tested per lot. A rigid control of temperature and other conditions was maintained.

Sugar flotation test. At least 400 peas from each lot were tested by the sugar flotation test developed at this Laboratory *(17)*. Whole peas were used with evacuation, and the temperature and other conditions were rigidly controlled.

Density. The average density of the peas was determined after peas were thawed in a covered dish over warm water, mixed and allowed to come to room temperature. This method, the details of which will be published later *(7)*, was based on the Archimedes principle with the elimination of air by evacuation from the peas and use of an 8% sugar solution.

Tenderometer values of cooked peas. The ordinary tenderometer pea scale can be used for the measurement of tenderness of cooked peas *(8)*. The method will be described elsewhere. Frozen peas were added to enough boiling distilled water to cover them and were cooked 8 minutes after resumption of boiling. The temperature of the peas used for the measurements was 23° C. ± 0.5° C. (73.4° F.). At least 3 and often 5 and 6 determinations were made for each average value reported.

Slurries for total solids and alcohol-insoluble solids *(4)*. About 100 g. of peas were blended with 100 ml. of 95% ethyl alcohol, transferred to a tared jar with 300 ml. additional alcohol, sealed, and the weight noted.

All sampling was done with a pipette which had its delivery end cut off to allow bits of tissue to pass without clogging. It was found very important to keep the slurry well agitated while samples were removed.

Total solids (T. S.). About 15 ml. of slurry was weighed in a tared dish, evaporated on a steam bath, and dried overnight in an air oven at 70° C. (158° F.) followed by 48 hours in a vacuum oven also at 70° C. The dry residue was weighed and the solids were calculated.

Alcohol-insoluble solids (A. I. S.). About 25 ml. of slurry was transferred to tared 50 ml. centrifuge tubes, weighed, centrifuged 3-5 minutes, decanted, and washed twice with 80% and twice with 95% alcohol with centrifuging, evaporated on a steam bath, and dried overnight in an air oven at 70° C. (158° F.) and for 48 hours in a vacuum oven at 70° C. The tube and residue were weighed and A. I. S. calculated.

Amylose index. Two amylose methods *(5, 11, 12)* were combined and modified. The solids from centrifugation of the alcohol slurry were solubilized in 53% perchloric acid, neutralized and the acidity adjusted with acetic acid. After dilution, potassium iodide and potassium iodate were added and the optical density determined. A reagent blank and a correction for turbidity were used. With no reference standard, this index is relative and was calculated as follows:

$$\frac{\text{Optical density} \times 100}{\text{Wt. of peas present in the final dilution}} = \text{Amylose index}$$

RESULTS

Table 1 summarizes the results of several comparative methods for measuring maturity in 4 varieties of frozen peas grown in 1951, 1950, 1949. Data in the table are arranged according to decreasing cotyledon texture scores, i.e., the organoleptic maturity of the peas increases from top to bottom for each variety and season.

After the harvest number and size group, the table gives the weight per pea, which reflects the variation in average pea size of the samples. Except for a few reversals, the tenderometer values on raw peas fall in the same order as the organoleptic scores.

The results of organoleptic appraisal appear next in the table. The cotyledon and skin texture scores range from 7 (very immature, tender) to 1 (very tough or mature). The actual range of maturity was greater in 1950 and 1951 than in 1949. The scores cannot be compared from season to season because of this difference in scales and also because the peas for each season were tasted separately.

Results of the brine and sugar flotation tests are presented in the tables as U. S. Grades for Frozen Peas and as percent sinkers in 13, 15 and 16% sodium chloride solutions and in the corresponding sugar solutions. The grades were based on the brine flotation test alone, as all the frozen pea samples had a green color, excellent flavor and were nearly free of defects. There is good agreement between the results of the 2 flotation methods, except for very mature peas in grades C and D.

The range of density values within each grade is very narrow. Table 1 shows that the density level which separates one grade from another is not a constant quantity, but varies somewhat with varieties and with seasons for the same variety.

Tenderometer values for cooked peas in Table 1 show a fair range and agree well with organoleptic results.

Columns 12, 13, and 14 in the table give the total solids, alcohol-insoluble solids and alcohol-insoluble solids as a percentage of dry weight. All of these show good correlation with cotyledon texture and skin texture results. The results of the 1949 measurements of the amylose index are given in the table, where a difference between the 2 varieties can be readily seen.

DISCUSSION

Organoleptic appraisal. The cotyledon and skin texture scores obtained from organoleptic appraisal of the frozen peas are considered as primary measures of maturity. Figure 1 shows the close agreement a, between cotyledon and skin texture scores, and b, between the scores of the 2 varieties of peas tested each season. The close similarity of the 2 organoleptic scores for a given sample reflect in part the difficulty of judging independently 2 or more quality factors in the same material. In any case, these similarities permit the comparison of any objective method with either of the 2 organoleptic measurements, i.e., the cotyledon or skin texture scores alone. Therefore, all subsequent comparisons of objective measurements were made against cotyledon scores only (Figure 1).

The scale used by our tasters is only a relative one. It changes from one organoleptic study to the next, and it may stretch or contract more at one end than the

TABLE 1

Objective and organoleptic measurements of maturity on 68 samples of peas

Harvest No.	Size group	Weight per pea g.	Tenderometer at 23° C. raw peas	Texture scores[d] Cotyledons	Texture scores[d] Skins	Flotation U. S. grade[e]	Flotation % Sinkers Salt	Flotation % Sinkers Sugar	Density at 26.7° C. g./cc.	Tenderometer at 23° C. cooked peas	Total solids %	Alcohol insoluble solids %	Alcohol insoluble solids % dry wt.	Amylose index
Variety Thomas Laxton 1951														
I	2	.21	75.2	6.9	6.9	A	0.0	0.0	1.051	—	17.1	8.0	47.0	—
II	2	.25	78.5	6.7	6.6	A	0.0	0.0	1.056	14.3	18.6	8.9	47.5	—
I	3 & 4	.35	77.8	6.5	6.3	A	0.0	0.0	1.055	19.8	18.5	9.2	49.6	—
II	3 & 4	.35	83.2	6.0	5.6	A	2.5	2.0	1.058	19.5	19.5	10.1	52.0	—
III	3 & 4	.37	87.7	5.3	5.1	A	0.5	0.5	1.064	21.9	19.7	11.2	56.7	—
I	5 & 6	.55	92.4	5.1	4.9	A	4.0	2.0	1.067	22.6	20.3	12.3	60.6	—
II	5 & 6	.57	108.2	3.7	3.4	B	4.0	4.0	1.076	42.4	22.6	15.5	68.6	—
III	5 & 6	.61	105.1	3.2	3.0	B	2.5	1.8	1.076	41.3	22.7	16.3	71.9	—
II	7	.74	124.2	2.6	2.4	C	6.0	5.8	1.083	50.6	24.1	18.0	74.6	—
IV	5 & 6	.60	125.9	1.8	1.7	C	5.3	7.0	1.086	57.1	25.0	19.3	77.2	—
III	7	.76	141.8	1.2	1.2	C	14.0	12.0	1.093	73.7	26.6	21.8	82.1	—
L. D. P = .05				.31	.32									
L. D. P = .01				.40	.42									
Variety Dark Seeded Perfection 1951														
VI	2	.17	78.7	6.8	6.6	A	0.0	0.0	1.042	—	17.1	8.5	50.0	—
VI	3 & 4	.33	94.0	5.0	4.9	A	3.5	4.2	1.066	25.4	21.6	12.9	59.7	—
V	3 & 4	.33	95.7	4.3	4.3	A	9.1	5.7	1.068	27.5	22.9	13.9	61.0	—
VII	3 & 4	.35	108.3	3.8	3.6	B	4.5	3.5	1.073	39.2	22.1	15.1	68.2	—
VIII	3 & 4	.34	116.1	3.0	3.0	B	7.5	4.5	1.076	42.2	23.2	16.6	71.4	—
VI	5 & 6	.46	121.1	2.6	2.6	B	7.5	8.1	1.084	52.7	24.8	18.3	73.6	—
V	5 & 6	.47	123.8	2.4	2.1	C	9.5	4.8	1.087	55.8	25.7	19.2	74.8	—
VII	5 & 6	.48	144.3	2.0	2.0	C	13.6	9.6	1.092	63.7	26.4	20.3	76.9	—
VIII	5 & 6	.49	148.2	1.4	1.5	D	18.9	14.0	1.093	67.3	27.7	22.1	79.8	—
L. D. P = .05				.31	.32									
L. D. P = .01				.40	.42									
Variety Thomas Laxton 1950														
I	2	.20	75.2	7.0	6.8	A	0.0	0.0	1.048	12.0	16.6	7.6	45.5	—
I	3 & 4	.33	77.3	6.4	6.2	A	0.0	0.0	1.055	14.3	18.8	8.9	47.5	—
II	3 & 4	.35	80.3	6.3	6.2	A	0.5	0.0	1.063	14.3	20.0	10.0	49.9	—
III	3 & 4	.36	84.1	5.6	5.6	A	2.5	3.0	1.064	15.6	20.8	11.2	54.2	—
I	5 & 6	.54	91.7	4.6	4.6	A	1.4	2.5	1.064	23.0	20.1	11.4	56.5	—
II	5 & 6	.51	90.5	4.6	4.5	A	5.0	5.0	1.071	27.5	22.6	13.2	58.2	—
IV	3 & 4	.36	95.1	4.6	4.4	B	2.5	2.5	1.066	29.9	21.4	13.1	61.0	—
III	5 & 6	.56	106.7	3.3	3.2	B	6.7	7.0	1.079	38.5	24.0	16.2	67.4	—
I	7	.72	115.8	2.8	2.7	B	2.9	4.0	1.077	—	23.2	15.8	68.2	—
II	7	.67	111.2	2.4	2.4	B	7.2	5.5	1.084	53.0	25.0	16.8	67.1	—
IV	5 & 6	.60	125.8	1.9	2.0	C	9.7	5.5	1.089	60.5	26.2	19.3	73.5	—
III	7	.71	136.8	1.4	1.4	C	8.4	6.0	1.089	69.8	26.4	20.4	77.1	—
IV	7	.70	139.3	1.3	1.4	D	18.1	15.5	1.096	71.5	27.7	21.2	76.7	—
L. D. P = .05				.26	.27									
L. D. P = .01				.35	.36									
Variety Oracle 1950														
V	2	.20	78.2	7.0	6.8	A	0.0	0.0	1.048	—	17.2	8.1	47.1	—
V	3 & 4	.30	88.1	6.4	6.3	A	0.0	0.0	1.057	13.9	18.9	9.7	51.3	—
VI	3 & 4	.34	84.0	6.1	5.8	A	1.0	0.5	1.056	15.4	18.9	9.8	**51.7**	—
VII	3 & 4	.35	**89.8**	5.9	5.5	A	4.0	3.5	1.054	16.7	18.4	10.2	55.2	—
VIII	3 & 4	.37	89.9	5.6	5.3	A	4.5	4.5	1.058	21.7	19.5	10.9	55.9	—
V	5 & 6	.50	**101.0**	4.8	4.6	A	1.0	0.5	1.069	23.8	21.5	12.5	58.1	—
VI	5 & 6	.52	107.4	4.0	3.9	B	3.0	1.7	1.070	33.8	22.2	14.6	61.2	—
VIII	5 & 6	.58	**136.2**	2.9	2.7	C	11.6	5.5	1.080	56.5	24.5	16.7	68.2	—
VII	5 & 6	.58	130.6	2.8	2.6	B	10.9	10.5	1.076	48.5	23.2	15.8	68.0	—
VII	7	.70	146.1	1.6	1.6	C	7.8	5.5	1.084	—	26.0	19.3	74.4	—
VIII	7	.71	145.7	1.5	1.4	C	10.4	9.0	1.085	—	25.7	19.2	74.7	—
L. D. P = .05				.26	.27									
L. D. P = .01				.35	.36									
Variety Thomas Laxton 1949														
I	2	.25	71.0	6.8	6.6	A	0.3	0.0	1.039	14.9	16.5	7.2	43.9	4.6
II	2	.25	73.8	6.8	6.5	A	0.0	0.0	1.037	16.8	17.3	8.2	47.3	5.3
I	3 & 4	.38	76.5	5.9	5.7	A	0.3	0.5	1.044	18.0	17.9	8.7	48.6	7.1
II	3 & 4	.38	79.5	5.7	5.4	A	0.0	0.0	1.046	18.6	18.4	9.4	50.8	7.7
III	3 & 4	.40	81.0	5.3	5.1	A	0.3	0.8	1.047	19.2	18.4	9.3	50.5	8.5
IV	3 & 4	.41	83.8	5.0	4.6	A	2.7	2.2	1.048	24.2	19.5	10.1	51.8	10.0
I	5 & 6	.56	89.2	3.9	3.9	A	3.0	4.0	1.059	25.1	19.9	11.5	57.7	11.6
II	5 & 6	.58	91.2	3.7	3.5	A	7.3	7.2	1.060	27.4	20.6	11.8	57.5	12.6
III	5 & 6	.60	101.7	2.8	2.7	B	3.7	2.8	1.067	38.8	21.4	13.3	62.0	16.4
IV	5 & 6	.60	103.0	2.4	2.1	B	4.0	4.0	1.067	40.4	22.6	14.2	63.0	17.5
III	7	.76	113.7	1.4	1.3	B	10.7	10.8	1.072	44.7	23.1	16.1	69.9	21.3
IV	7	.76	122.0	1.3	1.3	B	11.7	12.8	1.072	58.8	23.5	16.2	70.8	21.5
L. D. P = .05				.19	.20									
L. D. P = .01				.26	.27									
Variety Rondo 1949														
V	2	.25	69.1	6.8	6.7	A	0.0	0.0	1.038	15.1	15.6	7.2	46.2	5.7
VI	2	.25	72.8	6.8	6.7	A	0.7	—	1.040	16.0	16.4	7.9	48.3	7.0
V	3 & 4	.36	71.8	6.2	6.1	A	0.0	0.0	1.044	15.4	17.1	8.6	50.2	8.3
VI	3 & 4	.39	74.5	5.7	5.6	A	0.0	0.2	1.049	15.6	17.1	9.0	52.4	9.4
VII	3 & 4	.38	83.5	5.3	5.0	A	1.0	1.8	1.046	22.3	17.9	9.5	53.0	10.1
V	5 & 6	.51	83.0	4.9	4.8	A	0.0	0.2	1.055	18.9	18.4	10.0	54.2	11.3
VI	5 & 6	.55	85.8	4.6	4.6	A	2.0	2.5	1.054	18.0	19.6	10.7	54.5	13.4
VIII	3 & 4	.40	95.9	4.3	3.8	A	6.3	4.0	1.046	32.5	19.5	10.7	54.7	12.8
VII	5 & 6	.61	100.1	3.1	2.7	A	9.3	8.0	1.057	34.4	21.1	12.5	59.4	15.7
VII	7	.76	108.2	2.2	2.1	B	0.3	2.5	1.064	41.0	22.4	14.1	62.9	18.9
VIII	5 & 6	.68	115.6	1.8	1.8	B	5.0	4.2	1.064	55.9	22.9	15.0	65.6	19.3
VIII	7	.78	119.0	1.5	1.5	B	6.7	7.0	1.067	53.8	23.6	16.1	68.5	21.7
L. D. P = .05				.19	.20									
L. D. P = .01				.26	.27									

[d] Texture Scores
7 = very immature cotyledons, very tender skins
1 = very overmature cotyledons, very tough skins

[e] Based on flotation tests only.
Solutions of salt and sugar

Grade A	13%	20.6%
Grade B	15%	24.1%
Grade C	16%	25.7%

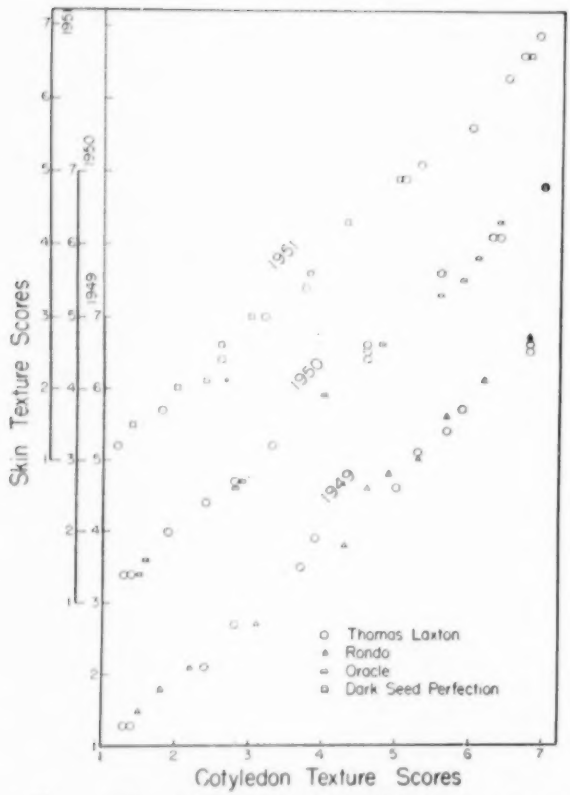

Figure 1. Comparison of skin and cotyledon texture scores in peas. Separate vertical (ordinate) scales are used for each year.

other. This is true in the present studies, as the whole scale of 1 to 7 was applied equally to different ranges of maturity. For example, score 2.4 was applied by the judges in 1949 to samples in grade B, while in 1951 the same score was used for samples in grade C. Therefore, here—as in most organoleptic studies—comparisons of actual scores should be limited to the samples tasted together *(2)*, i.e., only to the 2 varieties studied in one year.

As samples of comparable weight were used for most determinations, the greater the weight per pea, the higher the probable sampling error. The extreme sizes of 2 and 7 were studied in order to increase the range of maturities, although these sizes are seldom used for freezing. Moreover, the discrepancies due to the "pinning down" of the ends of the organoleptic scale in the instructions to the judges fall largely upon the least mature and most mature samples in each season's study, i.e., especially upon certain samples in the 2 and 7 size groups.

Comparison of objective with organoleptic methods.

Tenderometer measurements on raw peas (Table 1) and their relationship to cotyledon texture of the cooked frozen peas (Figure 2A) show that the tenderometer is a reliable tool for measuring maturity. This is true only when the limitations of this instrument are understood and the material carefully handled *(10)*. The same instrument was used throughout this study, with temperature of the peas controlled or corrected for and sampling carefully done and adequately replicated. Therefore the results agree relatively well with the organoleptic scores (Figure 2A). Tenderometer values for the cooked peas, included in Figure 2A for comparison, will be discussed later.

Varietal differences in the relationship of tenderometer values (raw) and cotyledon texture are not great, although in the 1950 season the panel rated Oracle peas as slightly more tender than Thomas Laxton peas for comparable tenderometer levels (Figure 2A). No such difference in these 2 varieties was observed with the other methods of measuring maturity.

The brine and sugar flotation methods for measuring maturity in frozen peas are based on the density. These and the direct measure of density suffer from the same basic weaknesses, i.e., the narrow range of densities involved, the vulnerability of the measurement to small changes in temperature and concentration of the liquid, the effect of any residual entrapped air, the influence of time as the peas absorb solutes and release water, thereby changing the density of both the peas and solution.

Compared to cotyledon texture scores, the U. S. Grades for Frozen Peas line up as expected. Occasionally, however, the percentages of sinkers in brine or sugar solution are not in the expected order, in spite of the fact that each value in the tables is based on a large sample.

In general, the flotation results obtained with skinned peas in brine agree with the results with whole peas in sugar solution after evacuation *(17)* although in 1951, Dark Seeded Perfection peas gave more variable results than Thomas Laxton peas, and in 1950 both varieties (Oracle and Thomas Laxton), showed greater variability than the varieties grown in 1949 (Thomas Laxton and Rondo). In this situation, not only variety but differences in growing season and maturity in 1950 and 1951 as compared with 1949 may account for some of this variability.

Of the 3 years studied, the density determinations of the 1951 season correlated best with cotyledon texture, possibly due in part to improved precision of the method (Figure 2B). The extreme precautions necessary for good results with the density method *(7)*, make it impractical for routine use. Varietal differences in density versus cotyledon texture, although small, appear to be present, especially at the higher maturity levels. The variety Rondo grown in 1949 also appeared to be more variable than Thomas Laxton for the same season.

Nevertheless, brine or sugar flotation methods are rapid and require little technical equipment or skill. Elementary care and large samples greatly improve the reliability of results. These methods are the only ones which separate out the most mature portion of a sample.

Figure 2A shows the agreement between the tenderometer value of cooked peas and their organoleptic maturity. While careful control of conditions is necessary, the method appears to be a reliable and practical one *(8)*. An improved and more compact shearing instrument, however, would make this method even more useful.

The relationships of percent total solids (T. S.) and percent alcohol-insoluble solids (A. I. S.) to cotyledon

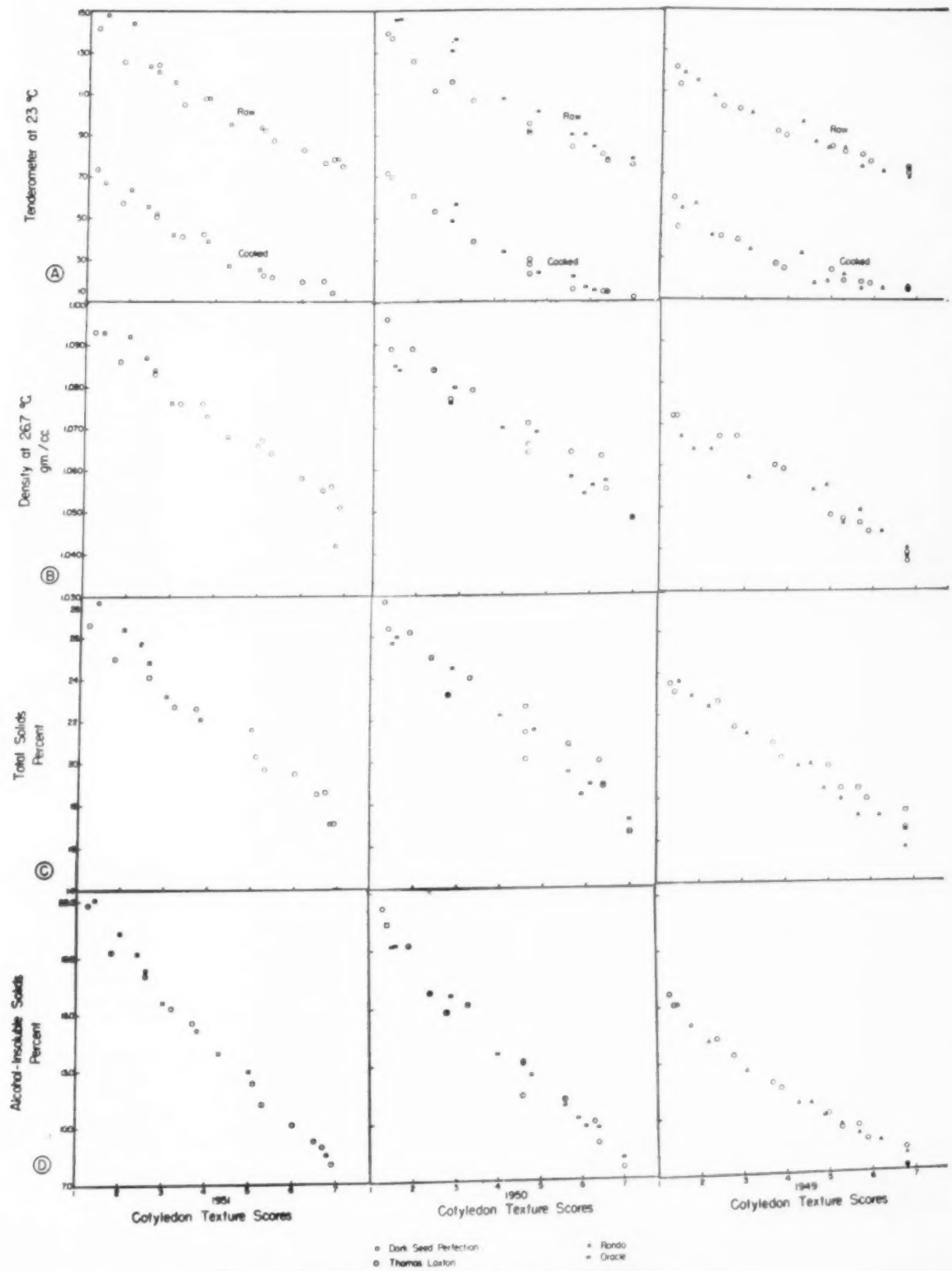

Figure 2. Comparison of objective tests and cotyledon texture scores:
A. Tenderometer values at 23° C. (73.4° F.) for raw peas and cooked (frozen) peas. B. Density in g./cc. at 26.7° C (80° F.). C. Percent total solids. D. Percent alcohol-insoluble solids.

texture scores are shown in Figures 2C and 2D, respectively. The percent of A. I. S. proved to be a better measure of organoleptic maturity than the percent T. S. The use of percent alcohol-insoluble solids based on dry weight (Table 1) has the advantage of being relatively independent of moisture losses often encountered in processing, storing and experimental handling of frozen peas. However, under the standardized conditions employed in these studies, this measure was not as good as the percent A. I. S. based on the weight of the frozen sample.

In view of the greater scatter of points in Figure 2C than in Figure 2D and of the good agreement between duplicate samples made from different samples of the original material, one must conclude that total solids is a poorer measure of maturity than alcohol-insoluble solids because of variations in the material itself. Perhaps this reflects fluctuation of sugar content due to weather conditions, time of day, temperature of harvest or post-harvest period *(6)*.

The amylose index (Table 1), though it increases rapidly with maturity of peas, was not determined in 1950 and 1951 because the 1949 results showed an appreciable difference between varieties. In addition to requiring considerable time and skill, this method appears to have no advantages over the determination of A. I. S. or T. S.

SUMMARY AND CONCLUSIONS

Organoleptic appraisals and a variety of objective tests were carried out on 68 samples of peas grown, harvested, and processed under carefully controlled conditions. These samples represented 4 freezing varieties, 3 seasons, and several harvest dates and size classes.

The design chosen for the organoleptic appraisals permitted the direct comparison of 20 or 24 samples in 6 tasting sessions. Good precision was obtained; statistical analysis showed that relatively small differences between average scores were significant.

The relationship between scores for cotyledon texture and skin texture for the samples of any one year showed a close correlation and was independent of variety. For this reason the cotyledon scores were taken as the primary standard of textural quality as it might appear to the consumer.

The percentage of alcohol-insoluble solids correlated more closely with cotyledon texture scores than did other objective measurements. These results confirm published evidence showing the high precision and reliability of alcohol-insoluble solids as a method for measuring maturity in frozen peas. Since this test is slow and somewhat laborious to carry out, a modification of it might be developed which would be more suitable for grading or routine determinations of maturity.

The density of the peas is difficult to determine precisely and even when so determined correlates only moderately well with the results of subjective appraisal. This fact places a limit on the reliability of flotation tests such as are now commonly used for grading commercial frozen peas.

The percentage total solids and the tenderometer value of the product after cooking also correlated moderately well with maturity as determined by human judges. The latter test is relatively rapid and deserves further investigation.

A test yielding an index of the amylose content of the peas showed a varietal difference in its relationship to subjective scores.

Acknowledgments

The authors are indebted to Professor Leonard H. Pollard and other members of the staff of the Utah State Agricultural Experiment Station for growing, harvesting, and shipping of the peas; to Harry M. Hughes and Terry A. Jeeves of the Statistical Laboratory, University of California, for suggestions regarding the analysis and presentation of data; to Ruth G. Witebsky and Elizabeth A. McComb for some of the analyses; to N. Floy Bracelin for preparation of the graphs; and to F. E. Lindquist, W. C. Dietrich, W. L. Stanley and many other members of the staff of the Western Regional Research Laboratory for help with the processing and for service on the taste panels.

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Abstracts

(Continued from page 14 preceding technical papers)

New growth factor discovered in liver.

Science News Letter, 62, 21 (1952).

Apparently, normal, healthy children fed a specially prepd. beef liver powder, made wt. and height gains of 40% and 20%, roughly more than those of the normal children without the liver prepn. The boys and girls were of av. height and their wt. was up to standard at the start of the test which lasted 13 weeks. The prepn. was given in the form of an orange flavored chocolate bar. The control children were given identical bars without the liver. The factor is not believed to be vitamin B_{12}. Further work on the distribution and nature of the apparently new factor is being done.

Chick-growth stimulation produced by surfactants.

ELY, C. M. *Science, 114,* 523-4 (1951).

Evidence collected to date on B_{12}-antibiotic-surface-active-agent supplementation, both alone and in various combinations, points to a similarity between the chick-grow response on antibiotics and surface active agents. Preliminary investigations of a possible synergistic effect between surfactants and B_{12}-antibiotic supplements have been negative.

The influence of ingested mineral oil upon the development of an essential fatty acid deficiency in the rat.

BACON, E. K. *J. Nutrition, 47,* 383-98 (1952).

Mineral oil, at levels of 2, 3, 5, 5, 7, 5 and 10%, was added to a fat-low diet which under ordinary conditions will result in a fatty acid deficiency and cessation of growth in 11 to 12 weeks. Below 5%, mineral oil did not result in cessation of growth earlier than 11 to 12 weeks, but the higher levels of mineral oil resulted in growth failure after a shorter period (two to three weeks on 10% mineral oil). In addn. to earlier cessation of growth, other symptoms of fatty acid deficiency appeared, including increased water consumption. The symptoms could be prevented by the inclusion of 50 mg. of linoleate per day. The fecal excretion of total fatty acids was found to be increased by the presence of mineral oil in the diet, and the iodine number of the fecal fatty acids was increased by mineral oil. A purely solvent action of mineral oil apparently cannot be accepted, however, since the deficiency symptoms were prevented by either oral or intraperitoneal linoleate.

Dextran consumed in body.

Science News Letter, 62, 91 (1952).

Dextran prepd. by fermentation from radioactive sugar is handled in the body and eliminated like carbohydrates. It does not necessarily give any appreciable amt. of nourishment to the body.

Dental caries in rats fed a diet containing processed cereal foods and a low content of refined sugar.

MCCLURE, F. J. *Science, 116,* 229-31 (1952).

A diet based upon cooked oat and corn grits induces caries in the rat which resembles the caries development in humans. This diet does not contain the coarse particles and is relatively low in sugars. Results suggest the possibility of a protein deficiency and caries development.

FOOD AND FOOD TECHNOLOGY

Chemical softeners for bread.

Food Drug Cosm. Law Repts., 98, 1, July 7, 1952.

The bread standards order previously reported at 2275 is stayed and the effective date postponed "until the conclusion of the review proceedings" in the United States Court of Appeals for the Third Circuit "insofar as the order relates to or prohibits the use, as an ingredient of bread, rolls, and other bakery products as designated in the order, of a polyoxyethylene monosterate product manufactured and sold by Atlas Powder Company under the trade name of Myrj 45." No written opinion was rendered by the court. The review proceedings are set for hearing during the week beginning October 6, 1952.

Beef "cocktail."

DRAUDT, N. *Food Eng., 24,* 67, June, 1952.

The product consists of a sterile, homogeneous liquid composed of meat and vegetables, sealed in a conventional 15-oz. can. It has tomato-juice consistency and light meat-gravy color. Among its inherent advantages are these: Flavor retention closely approximating that of cooked quality meat. Protein content per can in the range of one-third of man's daily need. Low caloric value—250-300 per can—suggesting ideal dietetic qualities. A satiating effect which should appeal to overindulgent eaters.

The keeping quality of whole milk powder. IV. The antioxidant properties of NDGA and certain emulsifying agents.

BUSCH, A. A., DECKER, C. W., AND ASHWORTH, U. S. *J. Dairy Sci., 35,* 524-32 (1952).

NDGA, alone and with added synergists, exhibited antioxidant activity when used in whole milk powder. The synergistic effect of ascorbic acid and citric acid with NDGA did not become noticeable until after about 9 mo. of storage at 85° F. The antioxidant activity of NDGA, alone and with citric acid as a synergist, appears to be manifested by a decrease in peroxide formation and an increase in ferricyanide-reducing values. Ascorbic acid added alone to whole milk powder at the rate of 50 mg. per liter of reconstituted milk did not exhibit any antioxidant activity. Certain polyhydric alc. esters and polyoxyethylene derivs. of stearic acid, when used in whole milk powder, appear to have antioxidant properties, as judged by results of organoleptic tests. The mechanism involved apparently is that of increasing the acid ferricyanide-reducing groups in the powder and decreasing peroxide formation. A surface adsorption film on the exposed fat surfaces may be indicated. They were not effective in preventing copper-induced oxidized flavor in pasteurized fluid whole milk.

Chicago ice cream industry markets vegetable fat frozen desserts in revolutionary development.

Ice Cream Trade J., 48, 24-5, June, 1952.

Ingredients are listed as being: water, sucrose, condensed skim milk, coconut oil, corn syrup, stabilizers and emulsifiers, salt, natural and artificial flavoring and certified color.

Inhibition of potato sprouting by 2,3,5,6-tetrachloronitro benzene and methyl ester of A-napthaleneacetic acid.

ELLISON, J. H. *Am. Potato J., 29,* 176-81 (1952).

Three separate though related tests are reported in this paper. It was found that 2, 3, 5, 6-tetrachloronitrobenzene (TCNB) was an excellent inhibitor to sprouting when used in bins of 10 or 25-bushel capacity, although it was a poor sprout inhibitor when used on lots of 10 or 12 potato tubers. TCNB was ineffective when treated tubers were exposed to free air circulation, as demonstrated by the excessive sprouting of tubers at the surface of treated bins. Methyl ester of a-napthaleneacetic acid (MENA) proved to be a good sprout inhibitor when used in either small or large lots of potatoes. Poor sprout inhibition resulted from the use of methyl ester of 2, 4, 5-trichlorophenoxyacetic acid (2, 4, 5-T).

Study of cause of texture change of canned salmon prepared from frozen fish.

Comm. Fisheries Rev., 14, 17, July, 1952.

Freezing and subsequent storage of salmon prior to canning produces adverse changes in the appearance and texture of the canned product. The most important changes are the formation of excessive curd and toughening of the canned product. Studies have shown that brining the cut sections of thawed salmon prior to canning reduces the extent of these changes. Statistical comparisons of the mean penetration values obtained from three canned salmon packs showed that canned salmon prepd. from frozen fish and packed in the normal style with dry salt is significantly tougher than the same fish if given a short brine treatment prior to canning. The fish used in these tests were coho salmon which had been held in cold storage at —20° F. for

(Continued on page 22)

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*Wetzel, N. C. et al.: Further Studies of Vitamin B_{12} Dietary Supplements, J. of Clinical Nutrition 1:17, Sept.-Oct. 1952.

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Abstracts

(Continued from page 18)

28 weeks before thawing and canning. The addn. of 2% disodiumphosphate to the brine did not appear to increase the efficiency of the brine treatment.

The measurement of lipase activity in oat products.

HUTCHINSON, J. B., AND MARTIN, H. F. *J. Sci. Food Agric., 3,* 312-20 (1952).

The lipase present in oats is only partially inactivated by the kilning process used for the manuf. of oatmeal, and the residual lipase sometimes causes considerable trouble because of its action in the meal when stored or when used in dough for oatcakes or biscuits. This hydrolytic activity can be almost completely eliminated in the mill by appropriate heat treatment but a rapid test was required for assessing the success of such treatment. A suitable method is described based on the rapid rates of fat hydrolysis found in doughs of 30-49% moisture content. A few meals show anomalous behavior when stored at 6-12% moisture content, but these can be detected by a modification which is suggested as a second and addnl. test for meals showing low but positive activity in dough.

Quarterly progress report; food irradiation and associated studies.

March 1, 1952 (NYO-3318); *Nuclear Sci. Abstracts, 6,* 532 (1952).

A report of progress in research on the radiation treatment of foods and biochem. materials, and certain related studies is presented. Previous work is reviewed briefly and modifications in the Co^{60} radiation source and technics for using it are described. The survival curve and sterilization dose for the mixed microbiol. population in raw, whole milk have been detd. Destruction of specific vitamns and phosphatase activity in whole and evapd. milk with increasing radiation doses is reported. These studies have also been extended to the Vitamin A in butter with similar results. Effects of radiation on isolated enzyme systems have been undertaken and the results of inactivation studies on catalase are reported. The radiation-induced reactions of fatty acids are being examd. but only limited effects are observed with considerable radiation doses. In order to det. more of the actual mechanisms by which nutrients are destroyed upon irradiation, the degradation of riboflavin is being followed in defined systems in order to gain some knowledge of the nature of changes induced and products formed.

High voltage removes filth.

ANDREWS, E. D. *Food Eng., 24,* 53-4, July, 1952.

A description of an electrostatic separator installed by Quaker Oats reported to remove all foreign matter including rodent excreta from corn. It is claimed that this method operates at close to 100% efficiency.

Medical aspects of mass dishwashing (conclusion).

CAIN, R. M. *Modern Sanit., 4,* 61-5, July, 1952.

This final paper in the series describes the methods used to make bacterial counts as a check on dishwashing operations. Methods of sanitizing utensils are discussed. Although chem. ultraviolet, or heat have been used, heat seems most practical.

Toxicity study of a spot fumigant.

ADAMS, E. M. *Modern Sanit., 4,* 39-41, July, 1952.

Dowfume EB-15, a "spot fumigant" used to control insects in food handling machinery, was investigated. This is a mixt. of carbon tetrachloride, ethylene dichloride, and ethylene dibromide. Based on acute vapor toxicity tests on rats, standards set for single exposures of workmen were not over 75 p.p.m. for 7 hr., 300 p.p.m. for 1 hr. and 1500 p.p.m. for 0.1 hr.

Technical facts for food processor on nickel and high-nickel alloys. Part I.

Food Eng., 24, 79-81, August, 1952.

Covered here are the general compn. and characteristics of the metals and their heat transfer rates. There is detailed information for tomato canners.

(Continued on page 24)

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Abstracts

(Continued from page 22)

Saline scramble.

Chem. Week, 71, 31-2, September 13, 1952.

A five year federal salt water purification research program is now being launched under auspices of Dept. of Interior. The aim is to bring costs of desalted sea water within the range of practical utilization by industry and agriculture in water-short areas. Two million dollars appropriated under a new law will finance the federal program.

PHYSICAL RESEARCH

The use of light scattering for determining particle size and molecular weight and shape.

Bender, M. *J. Chem. Education, 29,* 15-23 (1952).

Quant. information concerning size and shape of dispersed particles is obtainable by proper interpretation of the intensity and polarization of the light scattered from the soln. at given angles with the incident light. The conditions under which the Rayleigh, Debye, Mie or Cannabes concepts apply are discussed. The use of measurement of degree of depolarization is also discussed.

Recent developments in crystallization.

Bamforth, A. W. *Mfg. Chemist, 23,* 323-5 (1952).

Recent work in crystn. is reviewed. A method for the production of strong smooth crystals comprise distributing the supersatd. soln. in two zones, a crystg. zone for the mother liquor and a higher zone for growing crystals which can then be used as seed crystals in the lower zone. The effect of different rate of stirring upon crystn. and the effect of gelatin or agar-agar on dentritic crystn. has been studied. Other studies include the dielec. properties of the water of crystn. Production methods for the crystn. of ammonium sulfate, sugar and high polymers are discussed.

Principles and applications of ion exchange.

Nature, 170, 150-2 (1952).

Summary of papers presented at a symposium on the Principles and Applications of Ion Exchange.

PATENTS (U. S. A.)

Pudding compositions.

Kennedy, M. H., and Castagna, M. P. Assignors to Standard Brands, Inc. *2,607,692,* August 19, 1952.

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A SPECIAL REMINDER

IFT members are reminded that 1953 dues of $7.50 should be paid by January 31. The dues include a subscription to Food Technology; if you desire Food Research also, please add $5.50 to your remittance. Dues payments should be sent to:

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UTAH GETS FIRST FEDERAL-APPROVED FISH RESTORATION PROJECT

Utah has submitted the first project for approval under the terms of the new Dingell-Johnson Act granting Federal aid to the sport fisheries of the country, the Secretary of the Interior announced on September 24, 1951.

Utah's fishery project was received by the U. S. Fish and Wildlife Service on August 9, 1950, the same day President Truman signed the Dingell-Johnson bill, H. R. 6533, into law. The approval of this project, however, has been held up pending the final passage and signing of the Department of the Interior's appropriation bill for fiscal year 1952 which authorized the appointment of funds to the States for this new activity.

In its first project, Utah will seek data on how much public use is made of the waters of the State. These data can then be used to show the need for modification of fishing regulations and the inauguration of management measures, such as habitat improvement, in order to offer the best possible fishing and at the same time the proper protection to this natural resource. Opportunities for future development will be sought and the data obtained from field surveys will be used as a basis for developmental projects.

Utah proposes to find out the extent of the fish harvest taken by anglers, and the economic value of sport fishing to the welfare of the State. Although it is well known that supplying gasoline, boats, motors, tackle, bait, and guide service to fishermen has become a sizable business, Utah intends to find out the exact amount and its impact on the State's economy. The personal interview method, combined with questionnaire cards, will be used in the study.

Funds available under the Dingell-Johnson program are derived from the excise tax on sport fishing equipment, such as rods and reels, artificial lures, and fish creels. For each $3 allotted through the Federal Government, the State must contribute $1 of its own funds. The Fish and Wildlife Service, through its Branch of Federal Aid, administers the program, which includes the apportionment of available money to the States and the review and approval of each project to insure that it meets the standards set by Congress and the Secretary of the Interior. The Service also administers the outstanding successful Pittman-Robertson wildlife restoration program which has been operating since July 1, 1938. [From *Com. Fisheries Rev.*, **13** (9) 18 (1951) September.]

FATS AND OILS SITUATION IN 1951: A SUMMARY AND OUTLOOK

There was a strong demand from dealers and users in many countries in late 1950 and early 1951 for fats and oils to build up stocks, but this demand was apparently replaced in April-July 1951 by a tendency to reduce stocks. The world market price of most fats and fat-bearing materials rose about 80% from June 1950 to February-March 1951 but then declined. In late August and early September 1951, world market prices of fats and oils averaged about 25% above June 1950, the month of the outbreak of hostilities in Korea.

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World output of fats and oils had recovered by 1950 from the low levels of 1945 and 1946 to from 5 to 10% above prewar. With the increase of world population, however, production per person was still moderately below prewar.

Prospects in early September 1951 indicated a substantial increase in world production of fats and oils in 1951/52. A 75% increase in United States production of cottonseed is estimated this autumn. Production of groundnuts in West Africa and India and of olives in the Mediterranean area is expected to be larger than in 1950, a poor year for these crops. Production of lard, tallow, and greases in the United States and Western Europe probably will continue to increase in 1951/52.

Demand for fats and oils for industrial use and consumption as food in most countries of North America and Western Europe will remain strong in 1951/52 and may even increase, since consumer incomes and industrial activity are likely to be maintained or to rise still further. Demand for most fats and oils for stockpiling, however, will be considerably less urgent than in late 1950 and early 1951, barring another international crisis of the proportions of the Korean conflict. If the present good prospects for an increased output of fats and oils in 1951/52 are borne out and there is not another wave of scare buying, prices will average lower than in the 1950/51 marketing year. In the event of a recurrence of dollar-exchange difficulties, however, prices of fats and oils can be expected to decline less in European markets than in the United States, where the major part of the increase in the world exportable surplus in 1951/52 will be produced. (From FAO *Commodity Reports*, Fats and Oils No. 3, October, 1951).

HOTTEST FLAME CREATED FROM HYDROGEN AND FLUORINE

A flame said to be the hottest ever created by man—fluorine burning in hydrogen at 8000° F.—was described at the Temple University's Research Institute at a meeting celebrating the fiftieth anniversary of the National Bureau of Standards.

Comparing the flame directly with the sun, the research team found that at ordinary atmospheric pressure the temperature was 7000° F. (4300 K.) which increased to 8000° F. (4750 K.) when under five atmospheres pressure. By means of higher pressures the Temple University scientists expect to reach sun temperature (9000° F.).

The oxy-aluminum torch in previous Temple experiments melted anything against which it was directed, but the even higher temperature of the hydrogen-fluorine torch will likewise melt everything so far known on earth. The sun and the H-FL flame were directly compared by matching their brightness through glass filters by what is known as the line reversal method.

So far, the hydrogen-fluorine flame has no industrial use, since the combinations of oxygen with aluminum, magnesium, and zirconium provide sufficiently high heat for practical purposes. [From *Amino Laboratory News*, 9 (1) 1 (1952) January].

WORLD-WIDE PER CAPUT CONSUMPTION OF SUGAR

The following table shows not only the wide range of consumption per caput, but also the varying importance of sugar as a contributor to food energy in national diets. Especially noticeable is its relative insignificance in countries of the Far East. This is also still true for many countries in the Near East and in Africa. It may

Countries	Sugar consumption[a]		Calories from sugar as percentage of total calories	
	Prewar	Recent	Prewar	Recent
	Kg. per caput	*Kg. per caput*	%	%
United Kingdom	49[b]	37	17	13
Belgium-Luxembourg	28	31	11	12
France	24	23	9	9
Italy	7	11	3	5
Netherlands	33	37	12	13
Norway	37	23	11	8
Sweden	46	51	15	17
Germany (Fed. Rep.)	24	27	9	10
United States	49	49	16	16
Canada	48	52	16	17
Argentina	27	35	10	12
Brazil	25	30	12	14
Chile	25	26	12	12
Egypt	14	14	6	6
Union of South Africa	23	40	11	17
Australia	54	55	17	18
China	1	1	1	1
Ceylon	12	15	6	7
India	14[b]	12[b]	7	7
Japan	14	3	7	2

[a] Includes not only refined sugar but also glucose, honey, and syrups.
[b] Expressed as gur.

be roughly estimated that sugar in its varying forms provides not more than 7% of the total calorie intake for about half of the world's population, in contrast to figures ranging from 16 to 18% for countries like the United States, Canada, Sweden, Union of South Africa, and Australia.

[From *FAO Food and Agricultural Statistics*, **5** (1) 1952].

ALASKA SALMON CANNERY WASTE BEING USED AT FISH AND WILDLIFE SERVICE HATCHERIES

As a result of experiments carried out during the past few years at the Seattle Fishery Technological Laboratory of the Service's Branch of Commercial Fisheries, trimmings (formerly dumped at sea) from salmon canneries in Petersburg, Alaska, are now being shipped to Federal fish hatcheries in the State of Washington where they are used as a rich source of protein and vitamins in the diet of hatchery fish. The first shipment of 60,000 pounds of such material has already been received at the Federal fish hatchery at Leavenworth, Washington, and an additional 50,000 pounds will be delivered shortly.

In making possible this utilization of hitherto wasted material, several problems had to be overcome. Experiments at the Seattle Fishery Technological Laboratory developed a chemical preservative treatment whereby fish eggs can be preserved without refrigeration for limited periods, thereby permitting their collection at localities not possessing refrigeration facilities. As a commercial-scale test, a portion of the material being

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collected this summer is chemically preserved in this way and the remainder shipped frozen.

Another problem that had to be overcome was that of the development of a suitable shipping container. Use of tin containers was the only method permitted by commercial steamship company vessels and the high cost of shipping the empty tins to Alaska made such a packaging method prohibitive. A method of bagging the waste prior to freezing was developed, using an inner plastic bag and an outer burlap bag, the latter being somewhat smaller than the former and bearing the strain during freezing and handling.

Finally a practical method of collection of the desired trimmings at the cannery had to be worked out. A wooden chute was built under the iron chinks so that the soft visceral portions of the waste were collected and the bony collar portions, fins, and heads were discarded. The desired portions are being flumed to a draining table where they are drained and sacked.

Even though the bags are very roughly handled in transit, 950 bags have been delivered to the Leavenworth Hatchery in excellent condition and without the loss of a single bag.

Careful cost records of this collection of Alaska salmon waste are being kept. Preliminary indications are that this material may be delivered to ports on Puget Sound at a cost of about 5 cents per pound. In addition to use as a feed for hatchery fish, this material may find large markets as a feed for fur-bearing animals. [*Com. Fisheries Rev.*, **13** (9) 8 (1951) September.]

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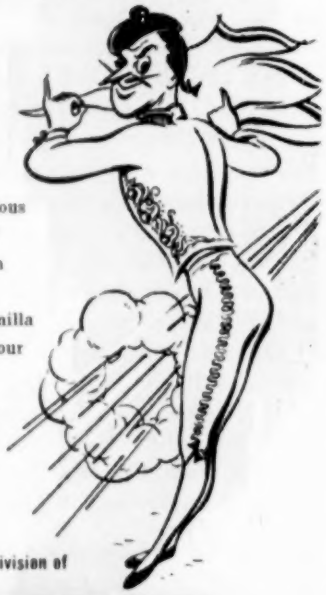

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FUTURE DATES FOR FOOD TECHNOLOGISTS

1953	
Jan. 22-23	National Wooden Box Association, St. Louis, Missouri
Jan. 26-28	National Dairy Council, Columbus, Ohio
Feb. 11-14	Institute of American Poultry Industries, Kansas City, Missouri
Feb. 19-20	Winter Meeting of the National Pickle Packers Association, Chicago, Illinois
Feb. 21-25	National Canners Convention and Canning Machinery and Supplies Association Show, Chicago, Illinois
March 1-6	National Frozen Food Convention, Chicago, Illinois
March 2-4	Quality Bakers of America, Chicago, Illinois
March 2-6	American Society for Testing Materials, Detroit, Michigan
March 8-11	American Institute of Chemical Engineers, Biloxi, Miss.
March 15-18	One hundred and twenty-third National Meeting of the American Chemical Society, Los Angeles, California
March 19-20	National Collegiate-Industry-Government Conference on Instrumentation, Michigan State College, East Lansing (Invitational: Address Electrical Engineering Dep't., Michigan State College, East Lansing, Mich.)
March 22-24	American Dairy Association, Chicago, Illinois
April 6-10	American Society of Biological Chemists, Chicago, Illinois
April 6-10	American Institute of Nutrition, Chicago, Illinois
April 20-22	American Management Association, National Packaging Exposition, Chicago, Illinois
April 20-22	National Frozen Food Industries Exposition, New York, N. Y.
June 21-25	**Thirteenth Annual Meeting of the Institute of Food Technologists, Boston, Massachusetts**

(Information concerning future dates of national and international meetings, conventions and exhibits of interest to food technologists should be sent to the editorial office.)

1951 FAO "YEARBOOK OF FOOD AND AGRICULTURAL STATISTICS" PUBLISHED

Volume V, Part 2, of this series brings up to date world trade statistics in food and agricultural commodities. The present 236-page, bound volume contains new figures for 1950 and revised data for the years 1947, 1948 and 1949, compared with the prewar (1934-38) average.

The general plan of the Yearbook follows lines of previous issues, with some minor changes in the presentation. Commodities have been grouped into sections, with summaries for some homogeneous groups (cereals, fruit, oilseed and vegetable oils, meats, textile fibers). The cereals section has been expanded with the addition of a table on trade in wheat and wheat flour, in terms of wheat, by crop year.

As a general rule, figures included in the tables have

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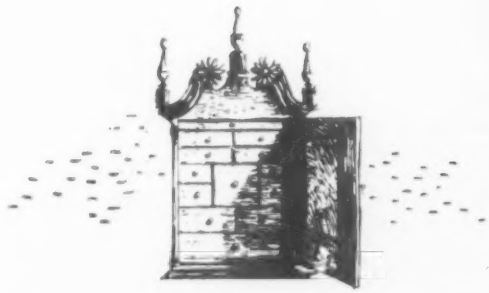
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been supplied by governments on special questionaires or through official publications. When official figures were not available, reliable unofficial sources have been used.

The Yearbook with its up-to-date statistics will be of interest to all students of international trade and of the world-wide food and agricultural situation. The 80 tables of data are followed by brief summaries entitled "Commodity notes" and "Country notes". The Yearbook is available through the sales agents for FAO publications, in the U. S. the International Documents Service, Columbia University Press, 2960 Broadway, New York 27, N.Y. The price of the present volume is $3.50.

U. S. CANNED FISHERY PRODUCTS PRODUCTION

The 1950 production of fishery byproducts in the United States and Alaska was valued at $76,218,997, 3% less than in the previous year. The principal byproducts were marine-animal oils, 21,763,849 gallons, valued at $17,472,709; marine-animal scrap and meal, 239,713 tons, valued at $29,225,928; marine pearl-shell, oyster-shell, and fresh-water shell products, valued at $16,312,004; and fish solubles, valued at $4,461,362. These byproducts were produced in 322 plants in 22 States and Alaska. [From *Com. Fisheries Review*, 13 (10) 11 (1951) October].

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AVAILABLE: FOOD TECHNOLOGIST, Ph.D. Background in agriculture, chemistry and bacteriology. 3 yrs. experience in production, food and dairy industries, wine-making. Desires position in research or production. REPLY BOX 233, Institute of Food Technologists, 176 W. Adams St., Chicago 3, Ill.

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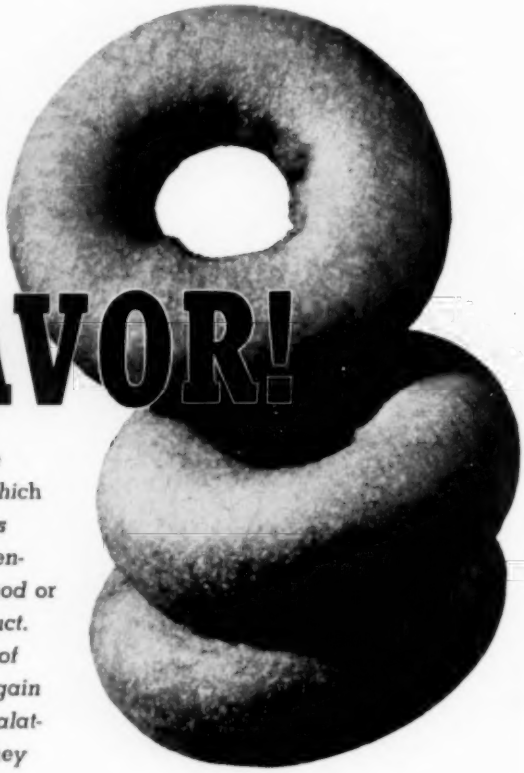

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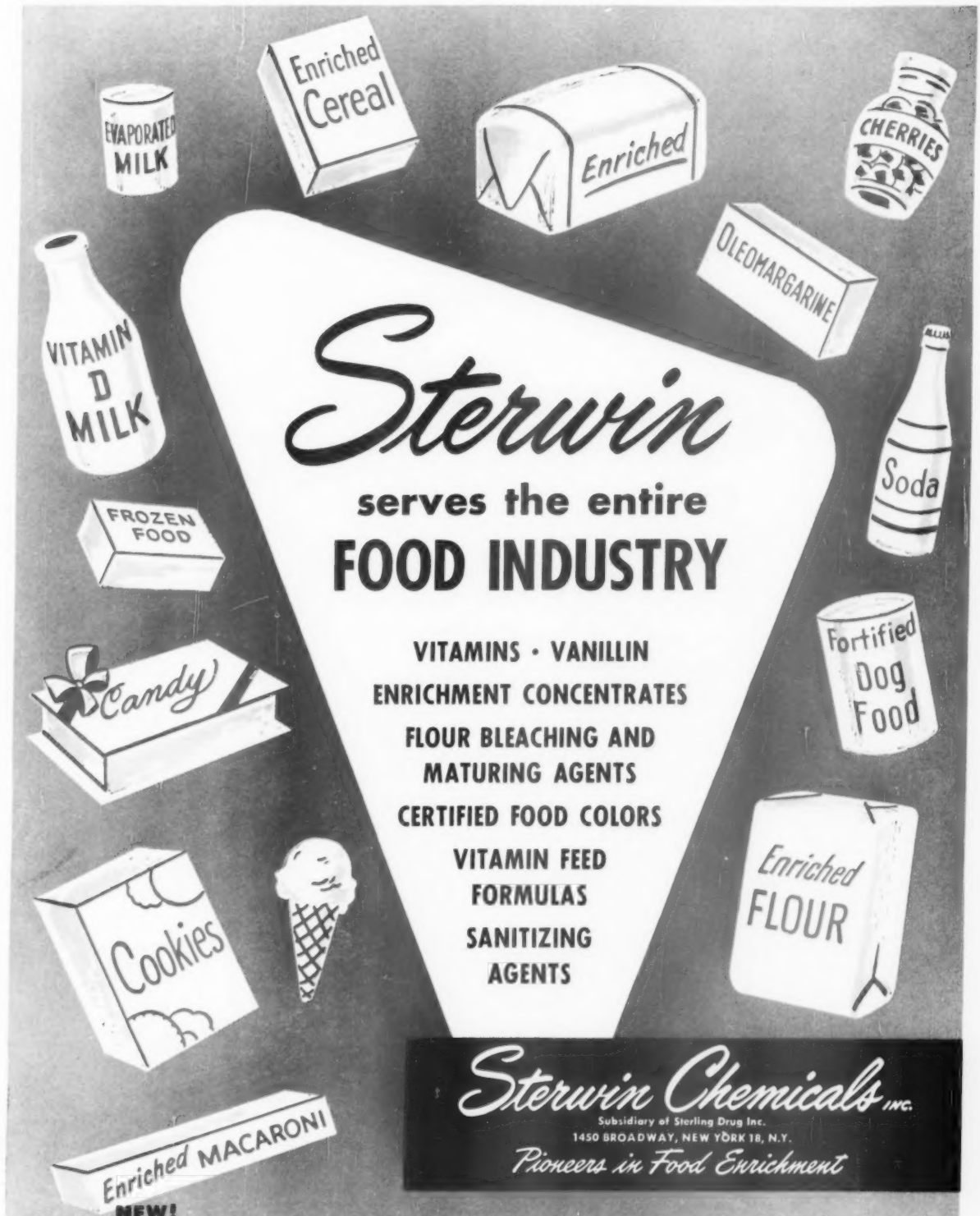

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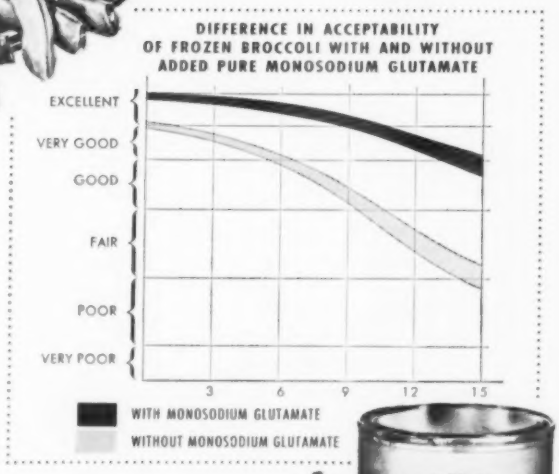

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